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DEVELOPMENT OF METHODS FOR DIMENSIONAL CHARACTERIZATION OF WET- AND DRY-MILLED CHRYSOTILE FIBERS

A Study of Cassiar Fibers

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MICROMEDIA

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PREAMBULE

This report describes the results of work initiated following contacts between CANMET, EMR (Ottawa) and C. Jolicoeur, PRAUS* (Sherbrooke) in September 1986. At that time, the main goals of the work were identified as the improvement of methods available, or the development of new methods, for the assessment of chrysotile fiber properties. Later discussions indicated a short-term need for the detailed characterization of fibers produced via wet-milling processes and extensive comparisons of wet- and dry-milled chrysotile fiber products. The research plan developed and executed under CANMET support (Department of Supply and Services, contract no. 23440-6-9205/01-ST) thus focussed mainly on these particular issues.

In the course of the work, a number of related issues arose which substantially broadened the scope of the project. Hence, the body of results described in the present report comprises two types of information: some are uniquely relevant to Cassiar wet- and dry-milled fibers, while others are of broader interest to the asbestos producers and user industries in general. We expect the latter type of information will be appropriately diffused and thus form the basis for further investigations of all commercially important Canadian chrysotile fibers.

Finally, as the reader will appreciate, the data reported herein rest on a wide variety of methodologies and expertises. Completion of this research was therefore possible only through collaborative efforts involving numerous scientists and laboratories: Don Doyle, CANMET, scientific authority assigned to the contract, J.-F. Alary, PRAUS* (general experimentation and data analysis), D. Nadeau and I. Lévesque, LCA**, PRAUS (microscopy and fiber counting techniques), M. Cossette

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* Programme de recherche sur l'amiante

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and G. Belleville, Ceram-sna (fiber opening and strength tests), M.-A. Simard, C. Théroux, Research lab. of C. Jolicoeur (fiber characterization), J. Khorami and A. Lemieux, LCA, PRAUS (fiber characterization), M. Taylor, W. Pratt, B. Talpin, Cassiar Mining Corp. (guidelines, fiber supplies and standard fiber tests).

EXECUTIVE SUMMARY

The following synopsis reports key observations, conclusions and recommendations issued from extensive physico-chemical investigations and standard tests carried out on several wet- and dry-milled chrysotile fiber products. For the most part, the results pertain to fibers produced by Cassiar Mining Corporation; for comparison purposes, selected results were obtained with other fiber products namely Woodsreef (Australia) and a Quebec 4T product.

While the project focussed mainly on methods for dimensional analysis of fibers, numerous other aspects of qualitative or quantitative fiber characterization were examined, particularly in relation to wet-milling processes developed by Cassiar Mining Corp. The following sections were thus organized to highlight distinctly the issues of broad interest to the Canadian asbestos industry and those of specific interest to Cassiar Mining Corporation.

1 - GENERAL GOALS

The overall goals pursued in current fiber characterization research in our laboratory are basically:

- The improvement of methods for characterizing the **dimensional** and **functional** properties of asbestos fibers.

- The development of **new approaches to fiber characterization** which could later be adapted for process monitoring and control in the asbestos mining industry.

2 - SPECIFIC OBJECTIVES

Within the framework of the present project, a set of specific objectives were defined concerning, first, a series of chrysotile fibers produced by Cassiar Mining Corporation and, second, selected aspects of fiber characterization of general interest to the Canadian asbestos industry.

2.1 - Specific objectives pertaining to Cassiar fibers

- Provide an extensive dimensional characterization of Cassiar wet-milled fibers, using standard testing methods and other direct size determination methods.

- Pursue a similar characterization of several grades of dry-milled fibers also produced by Cassiar.

- Pursue detailed comparison of the properties of wet- and dry-milled fibers.

- Provide specific recommendations for adequate dimensional characterization and quality control of wet-milled fibers.

- Explore methods for the direct determination of fiber surface area in aqueous slurries.

2.2 - Specific objectives of interest to the Canadian asbestos industry

- Provide a better understanding of the significance of standard test results (e.g., Bauer-McNett, fiber strength), through correlations of these results with the intrinsic properties of the fibers (e.g., lengths, diameters, morphology, surface areas, etc.).

- Evaluate the accuracy and reliability of a Fiber Length Analyser (Kajaani model FS-100) and its potential usefulness for length distribution measurements and quality control of asbestos fibers.

- Explore alternative methods for the "opening" of chrysotile fiber in aqueous slurries.

3 - RESEARCH STRATEGY AND WORK PLAN

The "Standard Chrysotile Tests" developed to provide the user industries with operational criteria comprises more than thirty (30) testing protocols grouped according to:

Dry fiber length (5 tests).
Wet fiber length (5 tests).
Openness, loftiness and drainage (7 tests).
Crude and granular content (2 tests).
Strength (3 tests).
Other physico-chemical properties (10 tests).

Although these tests fill an essential function, they provide little direct information on the basic properties which are responsible for the behavior of chrysotile fibers in industrial processes, and their performance in asbestos containing products.

3.1 - General requirements

In usual practice, the asbestos producers are concerned with fiber characterization at two levels: first, they require detailed characterization of their bulk fiber products; second, they require similar information on these fibers following extensive "opening" (e.g. fiber strength testing in A/C composites is performed after thorough fiber opening). Hence, a proper research strategy requires the detailed characterization of both the **bulk fibers** and the extensively **open fibers**.

With respect to quality control or process monitoring, the asbestos producers require characterization methods which will meet the following criteria:

- applicable to all fiber types;
- yield results relevant to user products or user processes;
- complement current standard tests or replace lengthly testing protocols;
- maximum ease and rapidity of methods;
- possibility of automation of readings or of entire testing procedure.

Research strategies designed to develop new characterization methods must obviously take these various requirements into account.

3.2 - Detailed strategies and work plan

3.2.1 - Dimensional characterization of fibers

In search of optimal dimensional characterization procedures, a detailed classification was developed of various methods which may be applied to provide information related to the size of the fibers. The methods envisaged were thus grouped in four classes (primary, secondary, tertiary and quaternary) as shown in Table 1.

TABLE 1: Classification of fiber size characterization methods

LEVEL	**METHODS**		
PRIMARY	OPTICAL MICROSCOPY (ℓ ; d)[1]	TRANSMISSION ELECTRON MICROSCOPY (ℓ ; d)	KAJAANI (ℓ)
SECONDARY	LIGHT SCATTERING (r_h)	NITROGEN SURFACE AREA (S.A.; d)	DYE ADSORPTION IN SOLUTION (S.A.; d)
TERTIARY	BAUER-McNETT [ℓ , r_h][2]	AIR PERMEABILITY [S.A.]	FIBER STRENGTH UNIT [ℓ ; d; S.A.]
QUATERNARY	FREENESS [S.A.]	ELUTRIATION [r_h]	

1 - Entries in parenthesis indicate the size parameters measured.
2 - Entries in brackets indicate size parameters upon which the results are believed to depend.
(S.A.: surface area; r_h: hydrodynamic radii)

Based upon this classification, and given the specific objectives previously outlined, the research strategy for adequate characterization of the Cassiar and other selected fiber products was fairly straightforward. The following work plan was thus outlined (as per contract):

A - Proceed to a detailed dimensional characterization of four grades of Cassiar wet-milled fibers using direct size measurement methods, namely: optical microscopy, transmission electron microscopy, fiber length analyzer (Kajaani), BET surface area, light scattering.

B - Proceed to a similar dimensional analysis of three dry-milled fibers chosen from groups 4 to 7.

C - For the same set of dry- and wet-milled fibers, carry out the following standard tests: elutriation, Bauer-McNett classification, Dyckerhoff air permeability, rapid surface area, freeness and fiber strength unit.

D - Through proper statistical analysis, correlate the size distribution data (ℓ, d, ℓ/d, surface area) of wet- and dry-milled fibers with the results of standard tests, in particular with Bauer-McNett classification.

E - Investigate dye adsorption methods for direct determination of the surface area of fibers in aqueous slurries.

3.2.2 - Other aspects

To complete the characterization of the fiber products investigated, numerous other physico-chemical tests were planned namely for: structural identification, chemical composition, brucite content, iron content, etc. To this purpose, chemical, thermogravimetric, X-ray diffraction and infrared spectroscopy analyses were also performed on the various fiber samples investigated.

Since, as mentioned earlier, we were further concerned with the properties of both "bulk" and "opened" fibers, various fiber opening methods were investigated, namely, turbine, ultrasonics and Dounce tube; the influence of the opening method on fiber properties were also examined

4 - MATERIALS

To provide relevant data on the various issues of the present investigation, the materials selected must comprise:

- wet-milled and dry-milled fibers;
- bulk wet-milled fiber products and wet-classified fiber groups;
- industrial dry-milled fibers and "opened" dry-milled fibers.

The various types of fibers selected for this work are listed in Table 2 below. Each fiber product was investigated first, as received (bulk fibers) and, second, after opening with a Pallmann mill operating at 6500 rpm. The Pallmann opening technique was used because of 1) its lower impact action compared to ball-milling and BOP treatment and, 2) its proven ability with Quebec fiber types to provide adequate opening for asbestos cement fiber strength tests. Several fiber products were examined after opening via alternative methods.

TABLE 2: Chrysotile fiber products investigated and their designation

FIBER TYPE	ORIGIN	DESIGNATION	FIBER, MILLING, OPENING
Wet-milled	Cassiar	FEED	bulk wet-milled fiber product
	"	S	shortest fraction isolated from FEED product by wet classification
	"	M	"Medium" fraction ..."
	"	L	"Long" fraction ..."
	"	XL	"Extra long" fraction ..."
	Woodsreef	10-mesh	wet-milled fiber product
Dry-milled	Cassiar	AX	bulk conventional dry-milled fibers
	"	AY	" "
	"	AZ	" "
	Quebec	4T	" "

5 - QUALITATIVE OBSERVATIONS ON CASSIAR WET- vs DRY-MILLED PRODUCTS

The physical aspects of wet- and dry-milled fiber products are nicely evidenced from optical or scanning electron microscopy. Typical photographs are reproduced in following pages. Additionally, we report in Table 3 below, various qualitative and quantitative results which illustrate some of the main features of the different types of Cassiar fibers examined. The "S" fraction of wet-milled fiber was consistently found to exhibit properties different from the other grades of wet-milled fiber, and thus justified a separate category.

TABLE 3: Selected properties of Cassiar dry- and wet-milled fibers

	Dry-milled (AZ, AY, AX)	Wet-milled (FEED, M, L, XL)	Wet-milled (S)
Bulk density[a] (lb/pi^3)	1.9 - 4.4	∿ 1.4	.7
Wet volume (cc)	500	∿ 1850	1925
Freeness (sec.)	45 - 113	126 - 171	442
Mag. rating	1.6 - 3.4	∿.25	.56
RSA (dm^2/g)	N.A.	∿190[a]	>260[a]
Surface area N_2 (m^2/g)	17	∿ 20	29.5
Surface charge (mv)	+27 to +31	+19	+19
Total iron content (as Fe_2O_3)	∿ 3%	<2%	2,1%
Brucite content[b]	undetectable	undetectable	undetectable
Carbonate (as CO_2)	<.1%	∿.4%	.53%
Fines (<200 Mesh)	24 - 38%	19 - 24%	71%
Non fibrous dust content[c]	low	very low	very low

a After opening
b From ATG curves
c From optical microscopy

The photographic observations and the data collected in Table 3 support the following general statements:

- The wet-milled (FEED, M, L, XL) fibers consistently appear "cleaner" than dry-milled fibers (lower magnetite, dust, and total iron content);

- The wet-milled fibers are more "opened" than their dry-milled conterparts as evidenced by their higher surface areas, freeness and wet volumes, and their lower bulk densities.

The microscopy results lead to other important qualitative findings, namely:

- From optical microscopy, the substantial -200-mesh fraction in the "S" grade is seen to consist mostly of short fibers; with little dust particles; (Figure 1);

- Electron microscopy reveals that the greater openness of wet-milled fibers compared to dry-milled fibers may be due to "loosening" of the fibrils in the individual bundles; the wet-milled fiber bundles frequently exhibit a "loosely bonded" appearance (Figure 2).

6 - FIBER LENGTH AND DIAMETER ANALYSIS THROUGH OPTICAL MICROSCOPY

The direct measurement of fiber lengths and diameters via optical microscopy at 2 μm resolution lead to detailed distributions from which average lengths ($\bar{\ell}$) and average diameters ($\bar{d}$) may be obtained, together with standard deviations (σ_ℓ, σ_d) on these quantities; low σ values refer to narrow distributions, thus to more homogeneous fiber sizes (note that under sampling and measurement conditions used in the present work, the coarse bundles, or long entangled fibers which would be found in the +4-mesh Bauer-McNett fraction were not measured; non fibrous dust was also ignored in microscopic examinations).

CASSIAR FIBERS

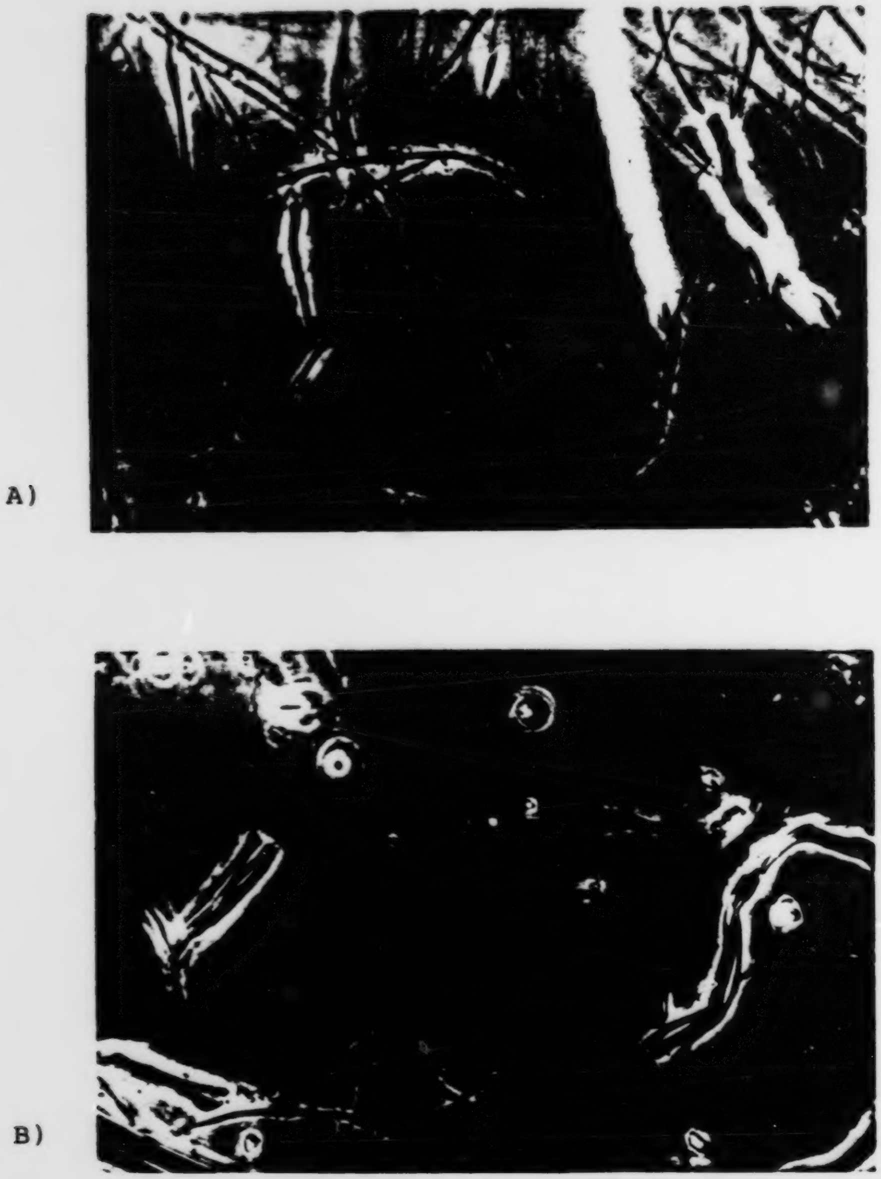
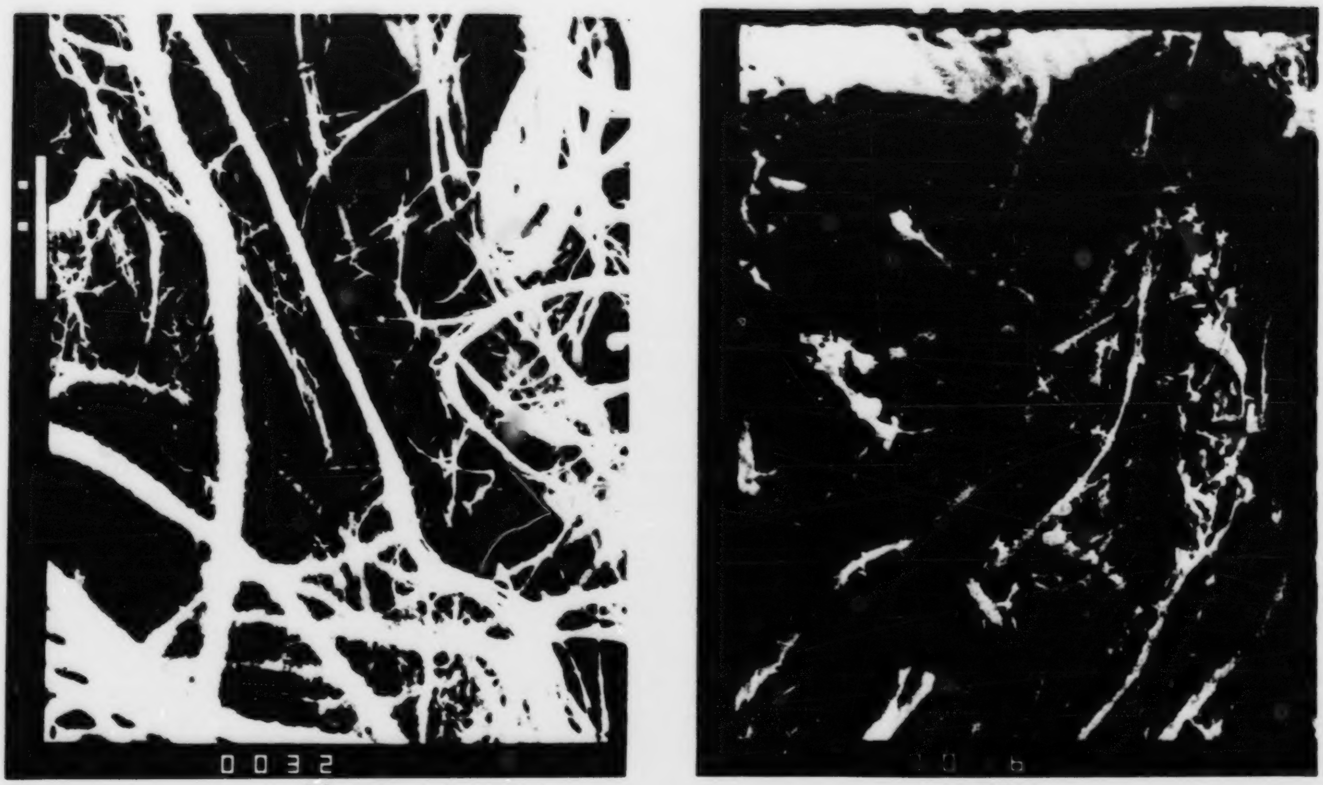


FIG. 1 Typical aspect of the fines in wet-milled (S) and dry-milled (AY) fibers under optical microscopy magnification at 540X magnification

A) Wet-milled "S" grade
B) Dry-milled AY product

CASSIAR FIBERS



A)

B)

FIG. 2 Typical aspect of bulk wet- and dry-milled fibers under scanning electron microscopy at 2100X magnification

A) Wet-milled "FEED" product
B) Bulk AY fibers

The key results derived from this analysis are collected in Table 4 which lists population averaged lengths and diameters for bulk and Pallmann opened fibers. Examination of these data points to the following:

- The average lengths ($\bar{\ell}$) of all dry- and wet-milled fibers, except the wet-milled S fraction, fall in the relatively narrow size range 0.35-0.8 mm;
- The same is true of the average fiber diameters ($\bar{d}$), although $\bar{d}$ values of bulk wet-milled fibers appear substantially lower than those of bulk dry-milled fibers;
- The homogeneity of fiber lengths and diameters, as reflected by the magnitude of the standard deviations σ_ℓ or σ_d, is generally greater for wet-milled fibers than for dry-milled fibers;
- Pallmann opening of the fiber products leads only to minor changes in their average lengths; the average diameters are reduced as expected, although with fibers having diameters in the .010 mm (10 μm) range, the average diameter is not significantly reduced upon Pallmann opening.

TABLE 4: Size distribution from optical microscopy of bulk and Pallmann opened fibers

	$\bar{\ell}$(mm)	σ_ℓ	$\bar{d}$ (mm)	σ_d
Dry-milled fibers				
Bulk				
AZ	.334	.530	.020	.025
AY	.467	.584	.025	.026
AX	.409	.589	.019	.021
Quebec 4T	.412	.658	.015	.012
Open (Pallmann)				
AZ	.421	.456	.019	.031
AY	.486	.626	.018	.028
AX	.557	.770	.014	.028
Quebec 4T	.443	.640	.015	.020
Wet-milled fibers				
Bulk				
S	.347	.257	.012	.007
M	.629	.482	.012	.012
L	.774	.552	.011	.013
XL	.749	.578	.010	.010
FEED	.624	.511	.018	.023
Woodsreef	.498	.597	.013	.013
Open (Pallmann)				
S	.380	.318	.011	.006
M	.596	.435	.012	.012
L	.666	.467	.014	.013
XL	.727	.532	.009	.009
FEED	.522	.408	.015	.012
Woodsreef	.443	.468	.011	.008

With regards to the bulk (FEED) and classified (S, M, L, XL) wet-milled Cassiar products, the following points are noteworthy:

- The average length of the "S" fraction is approximatively half that of the other wet-milled grades (M, L, XL or FEED), but the average diameters of S, M, L and XL fibers are nearly identical.
- The "S" fibers are substantially more homogeneous in both lengths and diameters than all other wet-milled grades (i.e., comparing σ_ℓ and σ_d for the various grades).
- The wet-milled Woodsreef 10-mesh fibers exhibit an average length intermediate between those of the Cassiar S and M products and a very similar diameter.

7 - FIBER LENGTH MEASUREMENTS WITH THE KAJAANI FS-100 FIBER LENGTH ANALYSER

Extensive experimentation with a Kajaani FS-100 Fiber Length Analyser yielded length distribution data which can be compared to results of optical microscopy and to data from standard classification tests. Typical length distributions (normalized) of Cassiar wet- and dry-milled fibers are illustrated in Figure 3. A detailed analysis of the results lead to the following conclusions:

- The Kajaani FS-100 instrument, although designed for wood pulp fibers, can measure the lengths of chrysotile asbestos fibers in the size range (approximate) 0.1-3 mm. (100-3000 µm); its resolution limit is 50 µm.
- The length distributions obtained with the Kajaani FS-100 analyser for either bulk or opened fibers were found to match fairly well those obtained by optical microscopy measurements especially with wet-milled fibers*. Within limitations given below, the FS-100

*** The mean length values computed by the FS-100 is only between 50 and 75% of what it should be, presumably due to some software problem; the length distribution data is however not affected.**

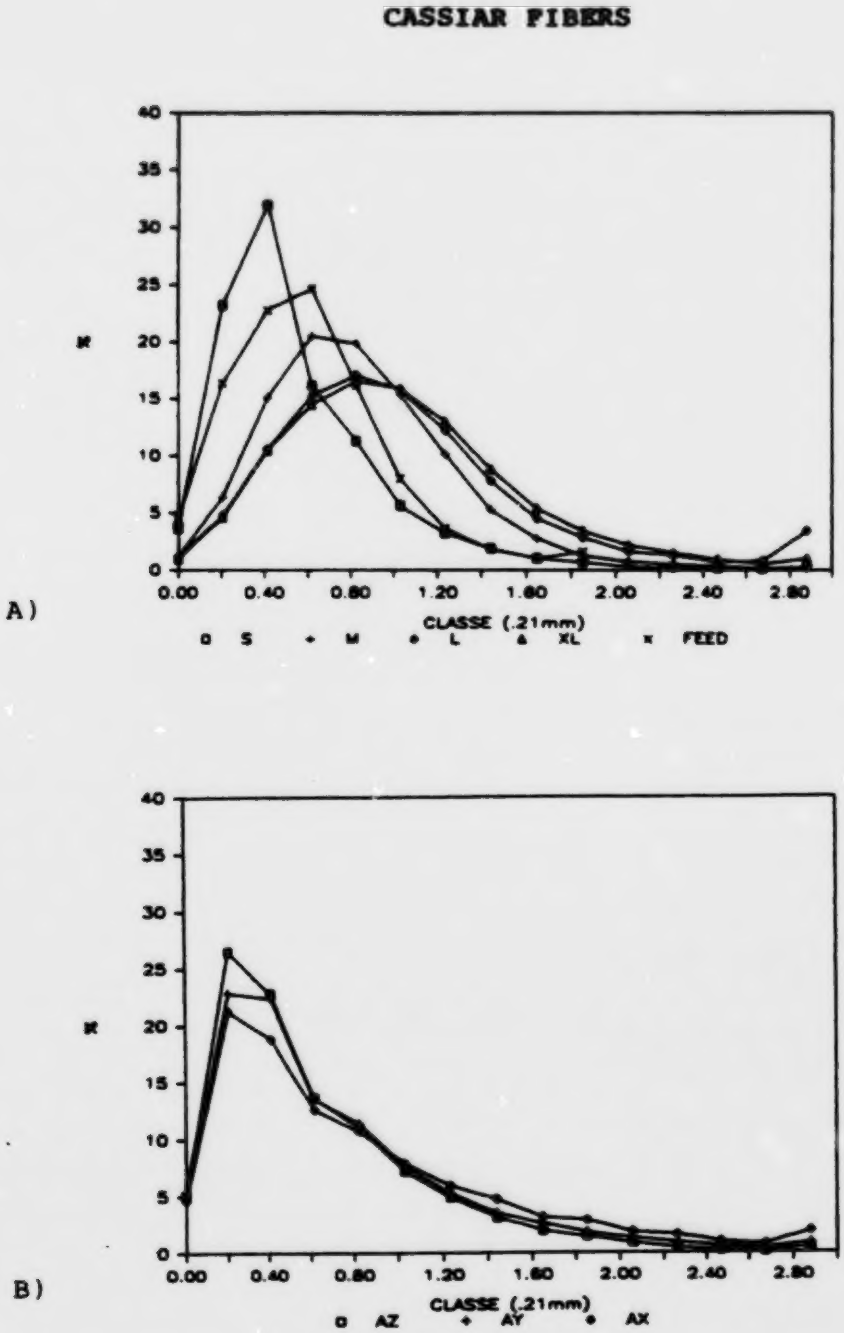


FIG. 3 Normalized fiber length distributions obtained for various Cassiar bulk fiber products using the Kajaani FS-100 Analyser

A) Wet-milled fibers: S: □ ; M: + ; L: ◇ ; XL: △ ; FEED: X

B) Dry-milled fibers: AZ: □ ; AY: +; AX: ◇

can thus rapidly (approx. 10 minutes) provide fiber length distributions of industrial chrysotile fibers with satisfactory reproducibility and precision.

- The main limitations of the Kajaani FS-100 were found to be:
  - frequent clogging due to larger fiber bundles, making the instrument highly unpractical with bulk fiber products especially dry-milled fibers; this problem is adequately alleviated by a prior mild opening treatment;
  - the FS-100 analyser slightly overestimates the short fiber groups. This can be understood as due either to the occasional counting of dust particles in the short fiber groups, or to the fact that the instrument "sees" only part of long fibers which are either too thin, curved, split, etc.;
  - the mean fiber lengths (population or normalized) computed by the Kajaani FS-100 is lower than it should if it were calculated as explained in the Kajaani literature (this is a computation or software problem and not one of the fiber measurement system);
  - since the Kajaani analyser does not yield information on the fiber diameters, evaluation of the weight fractions of fibers in the various length groups requires additional knowledge or assumptions regarding fiber diameters.

- The Kajaani FS-100 analyser thus appears ideal to characterize fibers having lengths which correspond to the Bauer-McNett fractions between +200 and -4-mesh (the Kajaani FS-100 "sees" virtually no fibers in the length groups corresponding to +4-mesh fractions).

- The routine use of a FS-100 analyser for quality control or monitoring will thus require independent measurements of the -200-mesh and +4-mesh fractions of the BMN classification (see below); the Kajaani FS-100 measurement would best work on wet-milled fibers having low dust contents.

8 - COMPARISON OF BAUER-McNETT CLASSIFICATION DATA WITH RESULTS OF OPTICAL MICROSCOPY AND KAJAANI ANALYSES

The comprehensive data sets available on fiber lengths and diameters from optical microscopy and from the Kajaani Fiber Length Analyser constitute an excellent basis for establishing the "true" dimensions of fibers within the various BMN fractions. Detailed analysis and comparison of Bauer-McNett data with direct size measurement results lead to the following conclusions:

- As expected, the average fiber lengths in the different BMN fractions do not correspond to the size of the screens appertures.

- The -200-mesh BMN fractions contain substantial quantities of fibers with lengths greater than 100 µm (200-mesh is equivalent to 70 µm); this is most obvious with the wet-milled Cassiar S fibers.

- the +4-mesh BMN fractions (the longest fiber group) contain considerable amounts of shorter fibers, presumably due to the formation of fiber aggregates (by physical clinging or adhesion in the aqueous suspension) which will not go through the 4-mesh screen.

- The optical microscopy data were used to calculate "effective" screen apperture sizes which would make the normalized OM weight distributions match the BMN results; this was not very practical, since the "effective" apperture sizes were found, as expected, to depend on the type of fibers.

- On the other hand, having noted that the average diameters of Pallmann opened fibers are relatively constant (only weakly dependent on fiber lengths), we used the Kajaani data on fiber lenght distributions to reconstruct BMN classification charts assuming:

  - constant fiber diameter (actual value is not relevant);

- BMN classes with cut-off lengths corresponding to the screen apperture sizes;
- neglect of dust content which would normally be included in the -200-mesh fraction.

The reconstructed BMN data are compared to actual BMN classification results in Fig. 4 and 5 for wet-milled and dry-milled fibers respectively.

- The agreement between the simulated and experimental BMN results cannot be quantitative since the Kajaani analyser does not measure dust particles or coarse fiber bundles; the comparison is still informative since it shows that, within a given BMN class, the relative order of the various fibers is reasonably well predicted.

Since the simulated BMN data were calculated from the Kajaani normalized length distributions, it would obviously be more incisive and easier to use these length distributions directly for fiber quality monitoring.

9 - FIBER STRENGTH TEST IN A/C COMPOSITIONS (FSU)

Fiber strength measurements[1] were carried out on the various fiber products after Pallmann opening at 6500 rpm. The fiber opening method was found adequate for the Cassiar wet-milled fibers, although it was later found that the Cassiar dry-milled fibers AZ, AY and AX required more extensive opening to achieve optimal FSU values. The key FSU results are collected in Table 5.

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[1] Experiments were carried out at Ceram-sna, Sherbrooke.

CASSIAR WET-MILLED FIBERS

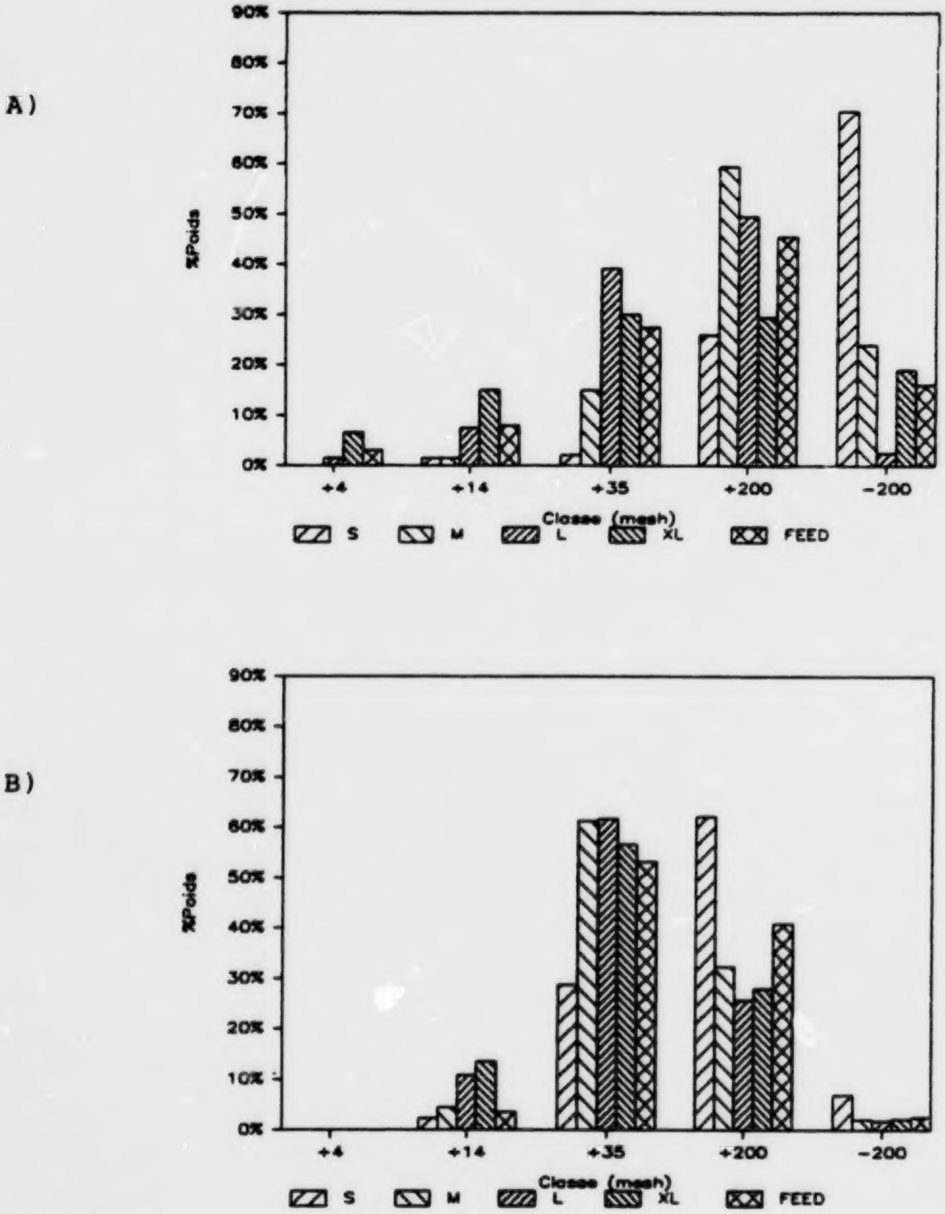


FIG. 4 A) Experimental Bauer-McNett results

B) Simulated Bauer-McNett results from Kajaani fiber length measurements

CASSIAR DRY-MILLED FIBERS

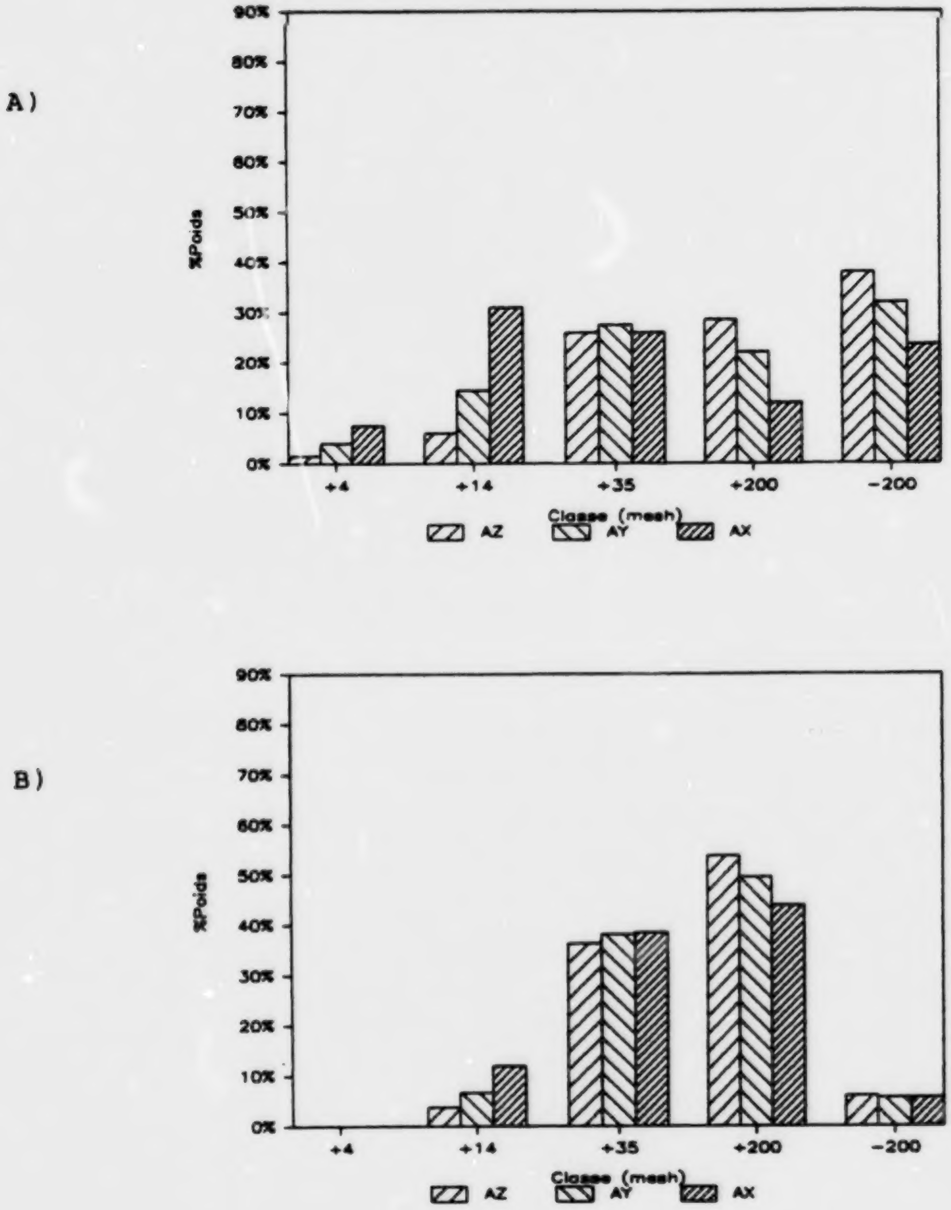


FIG. 5 A) Experimental Bauer-McNett results

B) Simulated Bauer-McNett results from Kajaani fiber length measurements

TABLE 5: Results of fiber strength unit (FSU) on Cassiar fiber products

	Opening technique	Bulk density (lb/pi^3)	Wt % of fiber in A/C mix	Adjusted rupture modulus (kg/cm^2)	FSU
Dry-milled fibers					
AZ	Pallmann	4.4	16.3	255.9	57.8
AY	"	2.7	11.4	261.7	84.0
AX	"	1.9	8.8	256.4	106.6
AZ	Ballmill & BOP		12.5	294.9	85.1
Wet-milled fibers					
S	Pallmann	0.7	4.3	186.8	---
S	"		6.0	188.7	---
M	"	1.3	6.8	251.7	135.4
L	"	1.5	7.6	262.8	126.2
XL	"	1.3	6.8	256.7	138.0
FEED	"		7.5	275.0	133.0
S	Turbine		6.2	202.6	---
M	"		7.4	278.5	136.7
L	"		7.9	254.4	117.8
XL	"		7.2	274.9	138.8

The FSU data show the following:

- Under comparable fiber opening conditions, wet-milled fibers yield higher FSU values than dry-milled fibers of similar average lengths; this is likely due to the higher degree of opening of wet-milled fibers: after Pallmann treatment, the average diameters of wet-milled fibers is approximately 50% lower than that of dry-milled fibers.

- All wet-milled fibers except the S group exhibit excellent cement reinforcement properties (FSU between 120 and 140) with either Pallmann (dry) or turbine (wet) fiber opening procedures.

- The S-type fibers impart good flexural reinforcement to cement at low fiber content; however, the rupture modulus (MRA) remains low (180-200) even when the fiber content is increased. This prevents the calculation of a significant FSU value.

- The classified wet-milled fibers (M, L, XL) yield similar FSU values; this is consistent with the fact that the M, L and XL fiber fractions have very similar average lengths and diameters.

- FSU values found with the bulk wet-milled fibers (FEED) are comparable to those of the classified M, L and XL fibers, suggesting that minimal classification is required for A/C application.

- With the S, M, L and XL fibers, the wet turbine opening method leads to FSU results comparable to those obtained after Pallmann (dry) opening.

10 - ALTERNATIVE FIBER OPENING METHODS

For most of the fiber opening requirements throughout this work, the Pallmann technique was used and the influence of Pallmann opening was examined in details. Alternative opening methods were also examined with the following general conclusions.

10.1 - Pallmann opening

It was generally found that Pallmann opening of bulk fibers at 6500 rpm lead only to a slight decrease in average lengths (∿ 10%); reductions in average diameters were also noted, which are most significant for fibers having diameters greater than 0.01 mm (or 10 μm). This finding, together with detailed microscopic examination of the fibers, before and after Pallmann treatment, led us to the following conclusions:

- under conditions used here, Pallmann opening does not produce substantial fiber opening when fiber diameters are less than $\underline{10}$ μm;

- Pallmann opening yields fibers which frequently exhibit "bruises", "kinks" and "end-splitting". These features contribute to reduce the bulk density and would tend to increase the apparent average diameter; they are also very likely to play an important role in cement reinforcement (i.e., providing additional anchoring points for cement particles) (Figure 6);

- with Cassiar dry-milled fibers, a single Pallmann treatment did not yield optimal fiber opening for cement reinforcement.

10.2 - Turbine opening

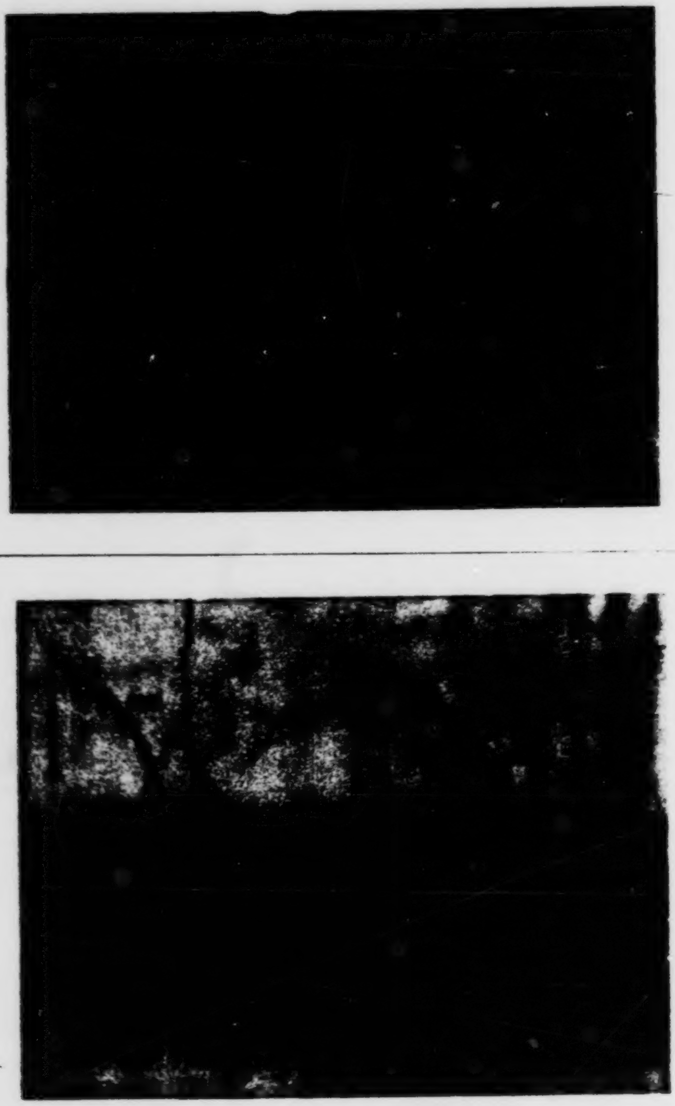
The extended high speed stirring of aqueous slurried in a turbine-type vessel (container with baffles and impeller stirring rod) yielded substantial fiber opening without significant reduction of average fiber length. The cross-shearing action exerted on the fiber in a wet turbine is weaker than in a Pallmann mill, so little fiber "kinking" or "bruising" was observed.

10.3 - Dounce tube opening

Fiber opening obtained by forcing an aqueous fiber slurry between the wall of a cylinder and a spherical-shaped piston yields highly opened fibers. The principle exploited in the Dounce tube is the same as that of the Szego Mill. Detailed length and diameter comparison show a reduction of average diameter greater than that obtained from Pallmann opening, but no significant reduction of average length.

The Dounce tube is a small scale laboratory method which would appear ideally suited for fiber treatment prior to fiber length measurement on the Kajaani analyser.

CASSIAR FIBERS



a) 6500 rpm

b) 13000 rpm

FIG. 6 S fibers opened in a Pallmann mill at different velocities (magnification: 100X; bar: 0.2 mm)

10.4 - Ultrasonic methods

The extensive ultrasonic treatment of aqueous fiber slurries leads to simultaneous fiber opening and fiber breaking. In fact, extented high power ultrasonic irradiation leads to very thin fibers having average lengths in the range of 10 µm!

11 - SURFACE AREA METHODS

In relation to quantitative measurements of the degree of fiber opening, three methods were explored which provide informaiton on the fiber surface area and porosity:

- BET nitrogen surface area
- mercury porosity
- dye adsorption in solution.

Although our investigation was limited to a few fiber products (wet- and dry-milled, bulk and opened), several noteworthy results were obtained which, at this time, mainly show the way for promising directions for future work:

- The BET nitrogen surface areas of fibers follow trends similar to the RSA (rapid surface area) results; however, the contribution due to dust (i.e., -200-mesh) appears far more important in the BET values than in the RSA values (this may suggest an approach to dust measurement).

- The mercury porosity results show major differences between bulk and Pallmann opened fibers (about 90% of the pore volume is found for pore radii between 0.1 and 3 mm) and only minor differences between wet- and dry-milled fibers.

- Studies on the adsorption of an organic dye (Acid Black) on fibers in an aqueous slurry yield two types of quantitative information related respectively to the surface area and to the affinity of the dye molecules for the solids. Our results show conclusively that surface areas can be obtained from dye adsorption measurements directly on wet fibers; however, the relationship between dye adsorption results and fiber surface area depends on fiber type (i.e., varying affinity); hence, the method will require "calibration" for each specific type of fiber.

12 - PROMISING DIRECTIONS FOR FUTURE WORK

In view of the long term objective of this work, namely, the development of automated methods for physico-chemical characterization of industrial chrysotile fibers, the results of the work described here point to the following strategic steps (in series or in parallel):

- Carry out a similar study on a broader variety of dry-milled chrysotile fibers, first from Canadian producers and later with selected competing fibers.

- Pursue the quantitative studies on dye adsorption by fibers in aqueous slurries to develop an in situ method for measurement of surface areas of wet fibers.

- Investigate means for automated evaluation of dust (non fibrous particles) contents. For this purpose, differences between RSA values and BET nitrogen surface area should prove useful with dry fibers; with wet-milled fibers, differences in the affinities of fibers and dust particles for organic dyes should be investigated.

- Investigate methods to evaluate in situ the fiber content of an aqueous fibers slurry in a wet-milled circuit.

- Examine further the relationship between mercury porosity and the degree of openness of fibers (dry-milled, or dried after wet-milling).

- Continue the investigation on the use of microwave heating methods, both for the purpose of drying wet-milled fibers and also as pretreatment to mechanical opening.

- Investigate aqueous turbine-type fiber opening equipment for incorporation into wet-milling circuits.

- Pursue work on ultrasonic devices for fiber opening in aqueous slurries (assisted by dispersants which are not detrimental to asbestos-cement compositions).

- Carry out a more detailed analysis of the consequences of wet-milling on the contents of various contaminants found in chrysotile fibers (e.g., iron oxides, magnetite, carbonates, brucite, nemalite, etc.)

- In conjunction with the above, pursue pilot plant work to help develop a detailed automated, monitoring and control scheme.

SOMMAIRE EXÉCUTIF

Le résumé qui suit décrit les principales observations, conclusions et recommandations issues d'un important programme de recherche et d'essais standards effectué sur divers types de fibres de chrysotile produites par voies sèche et humide. La majeure partie des données de cette étude ont trait à des fibres produites par la société Cassiar Mining Corporation; pour fins de comparaison, certaines données ont également été obtenues avec d'autres fibres, notamment celles de la société Woodsreef (Australie) et une fibre du type 4T produite au Québec.

Bien que la recherche était orientée principalement vers les méthodes de caractérisation dimensionnelle des fibres, plusieurs autres aspects de la caractérisation qualitative ou quantitative de fibres ont été examinés, principalement en relation avec les procédés en voie humide développés par la société Cassiar. Les sections qui suivent furent donc organisées de façon à distinguer les aspects qui présentent un intérêt général pour l'industrie canadienne de l'amiante et ceux d'intérêt spécifique à la société Cassiar.

1 - OBJECTIFS GLOBAUX

Les objectifs globaux de la recherche en caractérisation de fibres présentement poursuivie dans notre laboratoire consistent en:

- L'amélioration des méthodes pour la caractérisation des propriétés **dimensionnelles** et **fonctionnelles** de fibres d'amiante.
- Le développement de **nouvelles approches à la caractérisation des fibres**, lesquelles pourront être adaptées à la surveillance et au contrôle automatisés des procédés dans l'industrie minière de l'amiante.

2 - OBJECTIFS SPÉCIFIQUES

A l'intérieur de l'étude décrite ici, un ensemble d'objectifs spécifiques furent définis initialement en relation avec, premièrement, divers types de fibres produites par la société Cassiar et, deuxièmement, certains aspects de la caractérisation de fibres d'intérêt général pour l'industrie canadienne de l'amiante.

2.1 - Objectifs spécifiques relatifs aux fibres Cassiar

- Établir une caractérisation dimensionnelle exhaustive des fibres Cassiar produites par voie humide, ceci en utilisant divers essais standards et d'autres méthodes d'analyse dimensionnelle.

- Effectuer une caractérisation semblable de quelques grades de fibres Cassiar produites par voie sèche.

- Comparer de façon détaillée les propriétés des fibres produites par voies humide et sèche.

- Fournir des recommandations spécifiques relativement à la caractérisation dimensionnelle et au contrôle de la qualité de fibres produites par voie humide.

- Explorer des méthodes pour la détermination de l'aire spécifique de fibres en suspension aqueuse.

2.2 Objectifs spécifiques d'intérêt pour l'industrie canadienne de l'amiante

- Fournir une compréhension plus approfondie des résultats d'essais standards sur le chrysotile (p.ex. la classification Bauer-McNett, le renforcement de l'amiante-ciment), à travers des corrélations de ces résultats avec les propriétés intrinsèques des fibres (p.ex. longueur, diamètre, morphologie, aire spécifique, etc.).

- Évaluer l'exactitude et la fiabilité d'un instrument (Kajaani modèle FS-100) pour la détermination de longueurs de fibres et son utilité potentielle dans la caractérisation dimensionnelle et le contrôle de la qualité des fibres d'amiante.

- Explorer des méthodes alternatives pour le défibrage du chrysotile en suspension aqueuse.

3 - STRATÉGIE DE RECHERCHE ET PLAN DE TRAVAIL

Les essais standards ("Standard Chrysotile Tests") qui fournissent à l'industrie utilisatrice d'amiante des critères opérationnels comportent plus de trente (30) protocoles que l'on peut regrouper de la façon suivante:

Mesure de longueur à sec (5 essais)
Mesure de longueur humide (5 essais)
Ouverture, densité et drainage (7 essais)
Contenu en spicules et granules (2 essais)
Force tensile (3 essais)
Propriétés physico-chimiques diverses (10 essais).

Bien que ces essais standards jouent un rôle essentiel, ils fournissent peu d'information directe sur les propriétés spécifiques qui régissent le comportement des fibres d'amiante en procédé industriel et leur performance dans des produits à base d'amiante.

3.1 - Contexte général

Dans la pratique courante, les producteurs d'amiante sont intéressés par une caractérisation des fibres à deux niveaux: premièrement, ils doivent pouvoir compter sur une caractérisation détaillée des fibres brutes (fibres industrielles à la sortie de l'usine); deuxièmement, ils leur faut également des informations semblables sur les fibres ayant subi une «ouverture» poussée (lors des essais de force tensile de l'amiante dans une matrice amiante-ciment, les fibres industrielles sont, au préalable, ouvertes de façon optimale). Ainsi, une stratégie de recherche adéquate nécessitera une caractérisation détaillée à la fois des fibres brutes et des fibres ouvertes, c'est-à-dire après défibrage exhaustif.

En ce qui concerne le contrôle de la qualité des fibres ou la surveillance des procédés d'usinage, les méthodes de caractérisation envisagées doivent pouvoir répondre aux critères suivants des firmes productrices:

- applicable à tout type de fibres;
- fournir des résultats pertinents vis-à-vis les procédés et produits des utilisateurs d'amiante;
- complémenter les méthodes d'essais standards utilisées couramment ou remplacer les protocoles d'essais trop longs ou trop laborieux;
- offrir facilité et rapidité d'utilisation;
- pouvant être automatisé, soit pour l'acquisition des données, soit pour la procédure complète de l'essai.

Toute stratégie de recherche visant le développement de nouvelles méthodes de caractérisation doit évidemment prendre en considération ces divers critères d'acceptabilité.

3.2 - Stratégies détaillées et plan de travail

3.2.1 - Caractérisation dimensionnelle des fibres

A la recherche de méthodes optimales de caractérisation dimensionnelle de fibres, nous avons établi une classification des diverses méthodes pouvant être utilisées pour fournir des données reliées à la dimension des fibres. Les méthodes envisagées ont été regroupées en quatre classes (primaire, secondaire, tertiaire, quaternaire) tel qu'illustré au Tableau 1.

TABLEAU 1: Classification des méthodes d'analyse dimensionnelle de fibres

NIVEAU	MÉTHODES		
PRIMAIRE	MICROSCOPIE OPTIQUE (ℓ ; d)[1]	MICROSCOPIE ÉLECTRONIQUE À BALAYAGE (ℓ ; d)	KAJAANI (ℓ)
SECONDAIRE	DIFFUSION DE LUMIÈRE (r_h)	AIRE SPÉCIFIQUE (A.S. ; d)	ADSORPTION SE COLORANT EN SOLUTION (A.S. ; d)
TERTIAIRE	BAUER-McNETT [ℓ , r_h][2]	PERMÉABILITÉ À L'AIR [A.S.]	FORCE TENSILE [ℓ ; d; A.S.]
QUATERNAIRE	DRAINAGE [A.S.]	ÉLUTRIATION [r_h]	

1 - Les données entre parenthèses indiquent les paramètres mesurés ou obtenus.
2 - Les valeurs entre crochets sont les paramètres dimensionnels dont dépendent les résultats.

(A.S.: Aire spécifique; r_h: rayon hydrodynamique)

En s'appuyant sur cette classification et compte tenu des objectifs spécifiques mentionnés précédemment, la stratégie de recherche pour l'étude des fibres Cassiar et des autres fibres sélectionnées se révèle relativement simple. Le plan de travail qui suit fut donc élaboré (selon les termes du contrat):

A - Effectuer une caractérisation dimensionnelle détaillée de quatre (4) grades de fibres Cassiar extraites par voie humide utilisant des mesures directes de taille, notamment: la microscopie optique, la microscopie électronique en transmission, un analyseur de fibres (Kajaani), l'aire spécifique BET et la diffusion de la lumière.

B - Effectuer une analyse dimensionnelle semblable de trois types de fibres extraites à sec choisies parmi les grades 5 à 7.

C - Pour l'ensemble des fibres mentionnées en A et B, effectuer une série d'essais standards: élutriation, classification Bauer-McNett, perméabilité à l'air ("Dyckerhoff"), l'aire de surface rapide ("rapid surface area"), vitesse de drainage et force tensile dans l'amiante-ciment.

D - Via une analyse statistique adéquate, établir les corrélations entre les données dimensionnelles des fibres (ℓ, d,ℓ/d, aire spécifique) des fibres sèches et humides avec les résultats des essais standards, en particulier avec les résultats de la classification Bauer-McNett.

E - Étudier les phénomènes d'adsorption de teintures organiques en vue de déterminer in situ l'aire spécifique des fibres en suspension aqueuse.

3.2.2 - Autres apsects

Afin de compléter la caractérisation des diverses fibres étudiées, plusieurs autres essais physico-chimiques ont été planifiés notamment en relation avec l'identification structurale, la composition chimique, la teneur en brucite, la teneur en fer, etc. des fibres. A cet effet, diverses analyses chimiques et thermiques, de même que des études par diffraction de rayons-X et spectroscopie infrarouge ont également été poursuivies sur les divers échantillons de fibres étudiées.

Puisque nous devions examiner en parallèle les propriétés des fibres brutes et ouvertes, nous avons également étudié quelques méthodes de défibrage, notamment la turbine, les ultrasons et le tube de Dounce; l'influence de ces méthodes de défibrage sur les propriétés des fibres a également été étudié.

4 - MATÉRIAUX

Afin d'obtenir les données pertinentes aux divers objectifs de notre étude, nous avons dû examiner les catégories de matériaux suivantes:

- fibres produites en voies humide et sèche;
- fibres produites en voie humide à l'état brut et après classification humide;
- fibres industrielles produites par voie sèche à l'état brut et après défibrage.

Les divers types de fibres sélectionnées pour la présente étude sont identifiés au Tableau 2 ci-dessous. Chacun de ces matériaux a été étudié d'abord tel que reçu (à l'état brut) et ensuite après passage dans un défibreur Pallmann opérant à 6500 rpm. Le défibreur Pallmann a été préféré à d'autres appareils en raison de 1) son impact plus faible sur les fibres comparé au broyeur à boulets et au broyeur BOP et, 2) son efficacité démontrée dans le cas des fibres de gisements québécois pour le défibrage des fibres dans les essais de résistance d'amiante-ciment. Quelques échantillons de fibres ont également été étudiés après défibrage selon d'autres méthodes.

5 - OBSERVATIONS QUALITATIVES SUR LES FIBRES CASSIAR SÈCHES ET HUMIDES

L'aspect physique des fibres Cassiar produites en voies sèche et humide est avantageusement illustré à l'aide de photographies obtenues en microscopie optique ou microscopie électronique en transmission. Des photographies types sont reproduites dans les pages qui suivent (Fig. 1 et 2). De plus, nous résumons au Tableau 3 ci-dessous divers résultats qualitatifs ou quantitatifs qui illustrent quelques caractéristiques importantes des fibres Cassiar étudiées. La fraction «S» des fibres obtenues en voie humide démontre des propriété systématiquement différentes des autres fibres humides et ainsi justifie une catégorie distincte.

TABLEAU 2: Types et désignation des fibres de chrysotile étudiées

TYPE	ORIGINE	DÉSIGNATION	FIBRE, EXTRACTION, OUVERTURE
Humides	Cassiar	FEED	fibres humides brutes
	"	S	fraction courte séparée des fibres brutes FEED par classification humide
	"	M	fraction "moyenne" ...
	"	L	fraction "longue" ...
	"	XL	fraction "extra longue" ...
	Woodsreef	10-maille	fibres humides brutes
Sèches	Cassiar	AX	fibres industrielles extraites à sec
	"	AY	" "
	"	AZ	" "
	Québec	4T	" "

TABLEAU 3: Quelques propriétés des fibres Cassiar humides et sèches

	Extraction sèche (AZ, AY, AX)	Extraction humide (FEED, M, L, XL)	Extraction humide (S)
Densité lâche[a] (lb/pi^3)	1.9 - 4.4	∿1.4	.7
Volume humide (cc)	500	∿1850	1925
Drainage (sec.)	45 - 113	126 - 171	442
Prop. magnétique	1.6 - 3.4	∿.25	.56
RSA (dm^2/g)	N.A.	∿190[a]	>260[a]
Aire spécifique N_2 (m^2/g)	17	∿20	29.5
Potentiel de surface (mv)	+27 to +31	+19	+19
Fer total (i.e. Fe_2O_3)	∿3%	<2%	2,1%
Teneur en brucite[b]	non détectable	non détectable	non détectable
Carbonates (i.e. CO_2)	<.1%	∿.4%	.53%
Poussières (<200-mailles)	24 - 38%	19 - 24%	71%
Poussières non fibreuses[c]	faible	très faible	très faible

a Après ouverture
b Des essais ATG
c De la microscopie optique

Les observations photographiques et les données rassemblées au Tableau 3 appuient les énoncés suivants:

- les fibres humides (FEED, M, L, XL) apparaissent systématiquement plus propres que les fibres obtenues en voie sèche (teneurs plus faibles en magnétite, poussière et fer total);
- les fibres humides apparaissent plus «ouvertes» que leurs analogues produites en voie sèche (tel que démontré par des valeurs plus élevées d'aire spécifique, de drainage, de volume humide et de densité lâche).

Les études par microscopie mènent également à d'autre observations importantes notamment:

- la microscopie optique permet d'affirmer que l'importante fraction -200-mailles des fibres du groupe «S» est constituée principalement de fibres courtes ayant peu de poussières non fibreuses (Figure 1);
- la microscopie électronique (Figure 2) révèle que le degré d'ouverture des fibres humides apparaît systématiquement plus élevé que celui des fibres sèches; ceci peut être dû à un relâchement des fibrilles qui constituent une fibre individuelle; dans les fibres produites en voie humide, les faisceaux démontrent fréquemment une cohésion plus lâche que les faisceaux de fibres sèches.

6 - ANALYSE DES LONGUEURS ET DIAMÈTRES PAR MICROSCOPIE OPTIQUE

La mesure directe des longueurs et diamètres de fibres par microscopie optique sous une résolution de 2 µm permet d'établir des distributions de ces paramètres et des valeurs moyennes de longueurs ($\bar{\ell}$) et diamètres ($\bar{d}$); les écarts types sur les longueurs et diamètres sont également calculés (σ_ℓ, σ_d) et reflètent l'étalement des distributions, donc le degré d'homogénéité des dimensions des fibres.

FIBRES CASSIAR

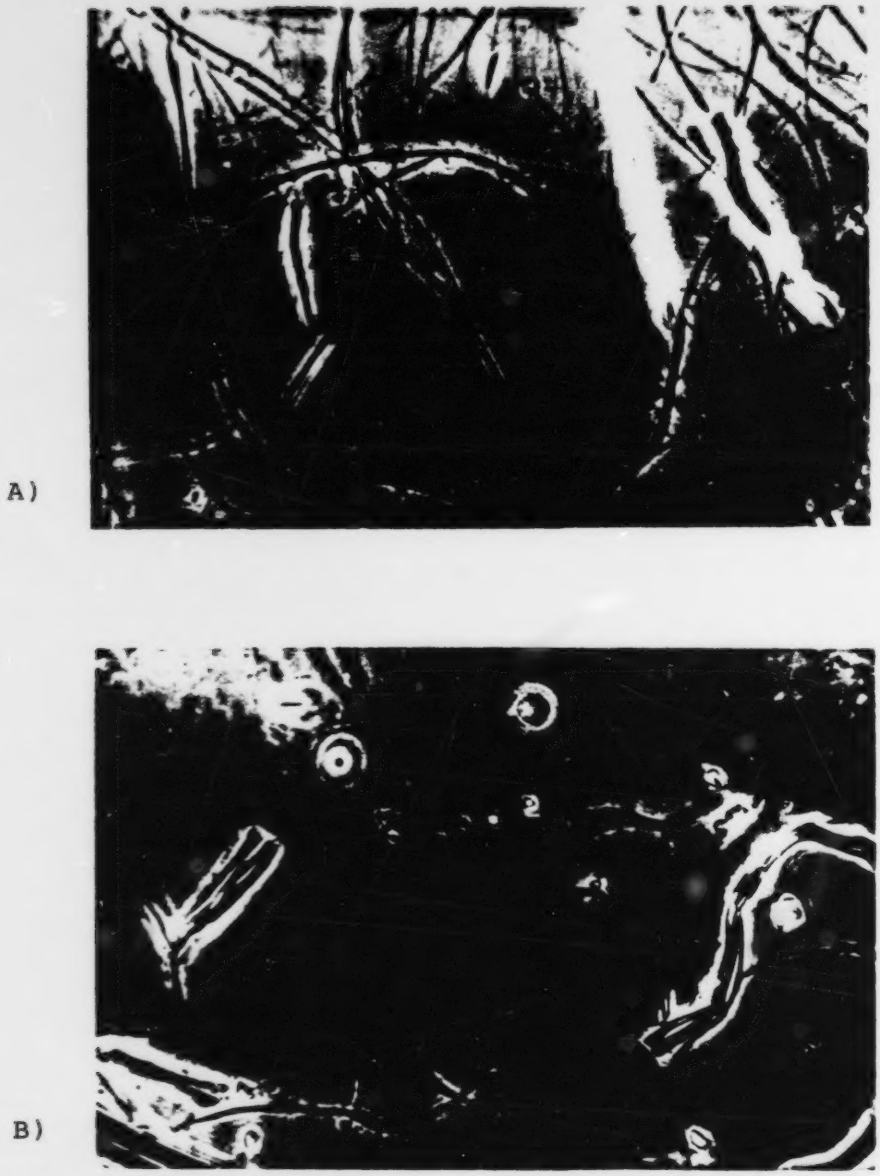
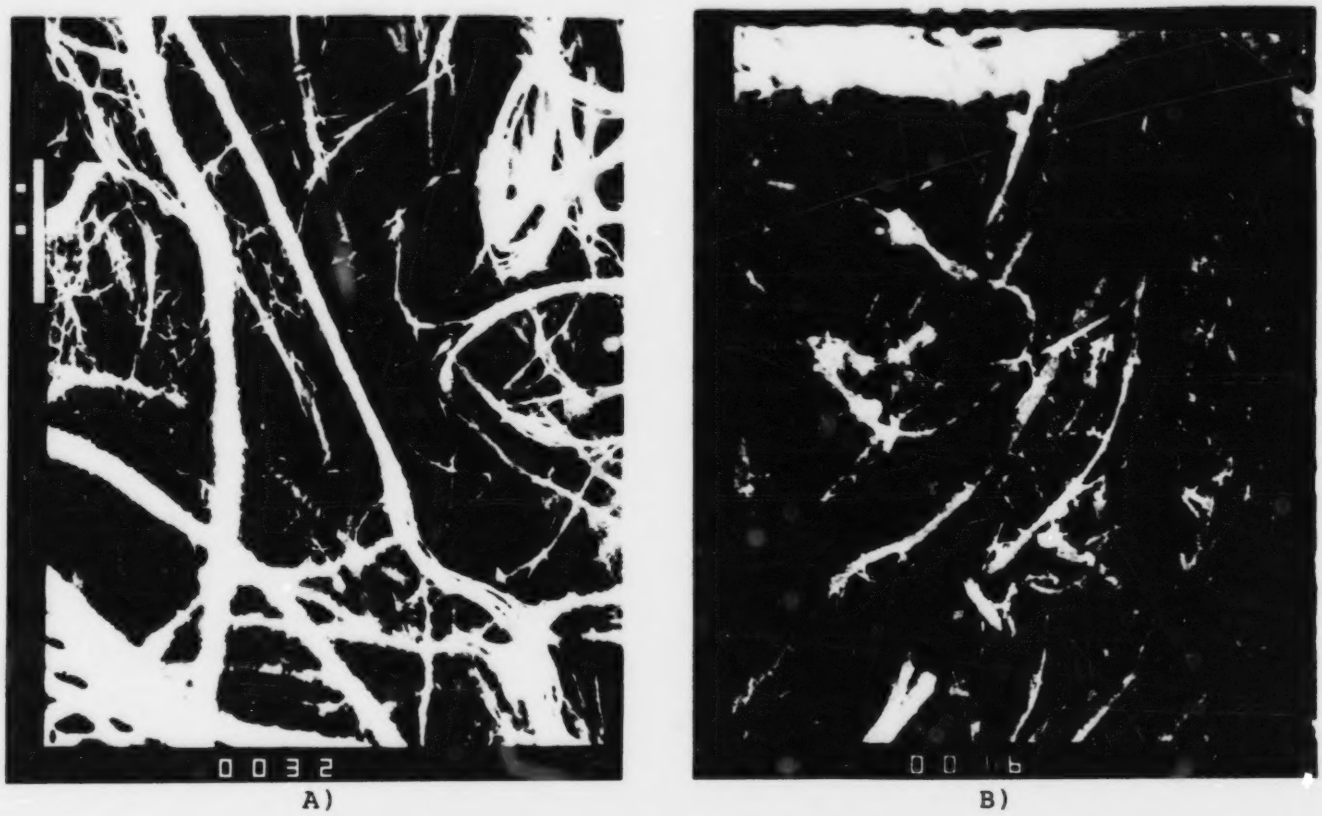


FIG. 1 Aspect courant des poussières et particules dans les fibres produites par voie humide (S) et par voie sèche (AY), tel qu'observé en microscopie optique sous agrandissement de 540X.

A) Fraction "S", voie humide
B) Grade "AY", voie sèche

FIBRES CASSIAR



A) B)

FIG. 2 Aspect courant de fibres produites par voie humide (produit brut) et par voie sèche (AY), tel qu'observé en microscopie électronique à balayage sous un agrandissement de 2100X

A) Produit brut, voie humide
B) Grade AY, voie sèche

Les principaux résultats issus de cette analyse sont rassemblés au Tableau 4 où nous reportons les diamètres moyens pour les fibres brutes et les fibres défibrées au Pallmann. L'examen de ces données révèle que:

- les longueurs moyennes ($\bar{\ell}$) de toutes les fibres étudiées (humides ou sèches), à l'exception de la fraction «S» des fibres humides, se situe dans un domaine relativement étroit entre 0.35 et 0.8 mm;
- il en va de même pour les diamètres moyens ($\bar{d}$), bien que, dans le cas des fibres brutes, les diamètres obtenus pour les fibres humides sont substantiellement plus faibles que ceux des fibres sèches;
- l'homogénéité des longueurs de fibres et des diamètres, tel que réflété par l'amplitude de l'écart type (σ_ℓ ou σ_d) est systématiquement plus grande pour les fibres humides que pour les fibres sèches;
- le traitement des fibres au défibreur Pallmann mène à des réductions mineures des longueurs moyennes; les diamètres moyens sont également réduits, bien que pour les fibres humides ayant un diamètre de l'ordre de 0,01 mm (10 µm), le diamètre moyen n'est pas réduit de façon significative suite au défibrage Pallmann.

En ce qui concerne les fibres brutes FEED et les fibres classifiées S, M, L, XL, les points suivants sont remarquables:

- la valeur moyenne des fibres dans la fraction «S» est approximativement la moitié de celle des autres grades de fibres classifiées en voie humide (M, L, XL ou FEED); toutefois, les valeurs de diamètres des fibres S, M, L et XL sont très voisines;

TABLEAU 4: **Distribution des tailles obtenues par microscopie optique pour des fibres brutes et ouvertes (Pallmann)**

	$\bar{\ell}$ (mm)	σ_ℓ	$\bar{d}$ (mm)	σ_d
Extraction sèche				
Brutes				
AZ	.334	.530	.020	.025
AY	.467	.584	.025	.026
AX	.409	.589	.019	.021
Québec 4T	.412	.658	.015	.012
Ouvertes (Pallmann)				
AZ	.421	.456	.019	.031
AY	.486	.626	.018	.028
AX	.557	.770	.014	.028
Québec 4T	.443	.640	.015	.020
Extraction humide				
Brutes				
S	.347	.257	.012	.007
M	.629	.482	.012	.012
L	.774	.552	.011	.013
XL	.749	.578	.010	.010
FEED	.624	.511	.018	.023
Woodsreef	.498	.597	.013	.013
Ouvertes (Pallmann)				
S	.380	.318	.011	.006
M	.596	.435	.012	.012
L	.666	.467	.014	.013
XL	.727	.532	.009	.009
FEED	.522	.408	.015	.012
Woodsreef	.443	.468	.011	.008

- la fraction «S» est considérablement plus homogène en longueur et en diamètre que tous les autres grades (comparer σ_ℓ et σ_d pour les divers grades);

- les fibres Woodsreef 10-mailles extraites en voie humide affichent des longueurs moyennes intermédiaires entre celles des fibres Cassiar «S» et «M» et leur diamètre moyen est très comparable à celui des groupes «S» et «M».

7 - MESURES DE LONGUEURS DE FIBRES À L'AIDE D'UN KAJAANI FS-100

Des essais systématiques à l'aide d'un analyseur de fibres Kajaani (FS-100) ont généré des distributions de longueurs de fibres qui peuvent être comparées aux résultats obtenus en microscopie optique ou aux données obtenues des essais standards. Des distributions types (normalisées) obtenues pour les fibres Cassiar extraites en voies humide et sèche sont illustrées à la Figure 3. Une analyse détaillée des résultats mène aux conclusions suivantes:

- Le Kajaani FS-100, bien que développé pour utilisation avec des fibres de cellulose, peut servir à déterminer la longueur de fibres de chrysotile dans le domaine (approximatif) 0.1 à 3 mm (100 à 3000 µm); sa limite de résolution est de 50 µm.

- La distribution de longueurs obtenues avec le Kajaani FS-100 pour des fibres brutes ou ouvertes est en accord satisfaisant avec les distributions obtenues par microscopie optique*. A l'intérieur de certaines limites décrites ci-dessous, le FS-100 peut donc rapidement (environ 10 minutes) fournir des distributions de longueurs de fibres industrielles de chrysotile selon une reproductibilité et une précision satisfaisante.

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* La longueur moyenne calculée par le FS-100 est entre 50 et 75% de ce qu'elle devrait être, probablement en raison d'un problème de logiciel; les données de distribution de longueurs sont toutefois correctes.

FIBRES CASSIAR

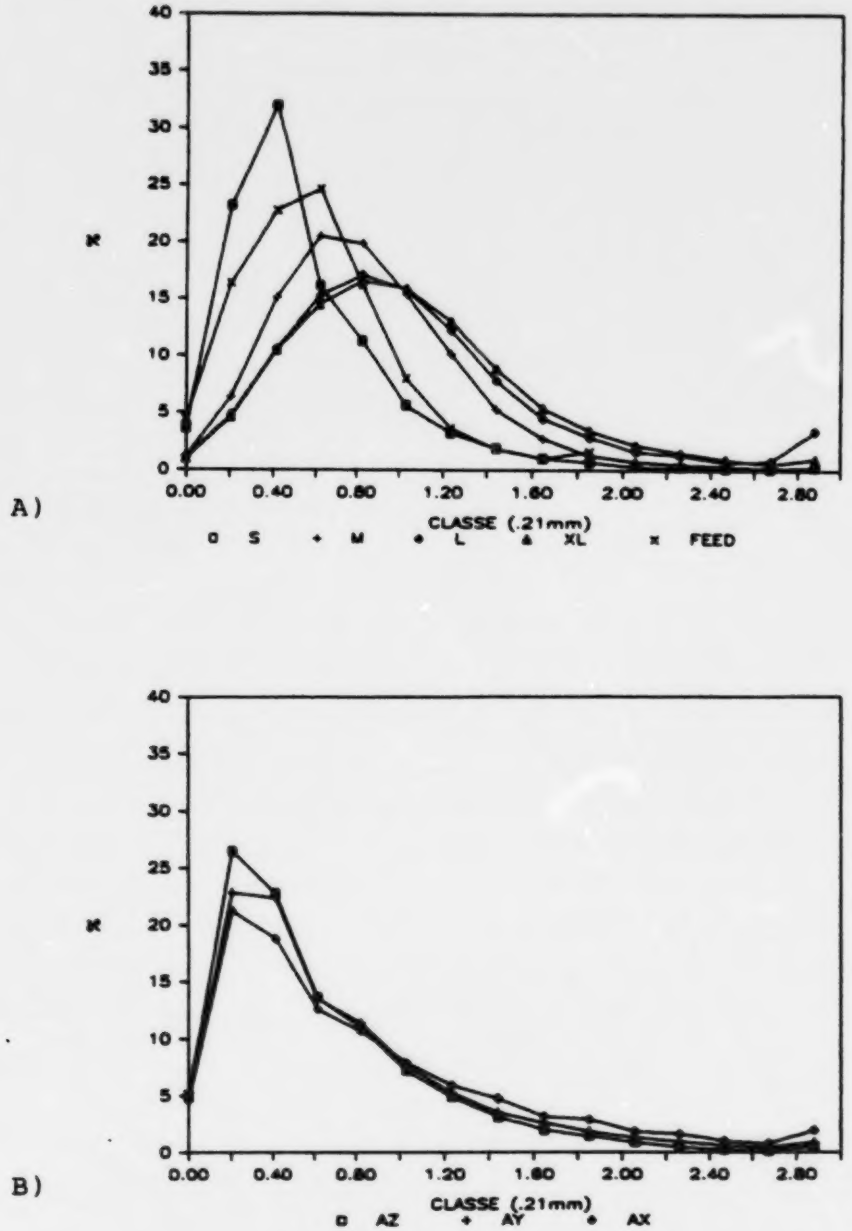

FIG. 3 Distributions normalisées des longueurs de fibres de différents produits de la mine Cassiar, tel que mesuré par un analyseur de longueurs Kajaani FS-100

A) Fibres produites par voie humide: S: □ ; M: + ; L: ◇ ; XL: △ ; FEED: X

B) Fibres produites par voie sèche: AZ: □ ; AY: + ; AX: ◇

- Les principales limites de l'appareil Kajaani FS-100 se sont avérées:
  - le bloquage fréquent du capillaire dû à des faisceaux grossiers ou granules, rendant l'appareil peu pratique pour utilisation avec des fibres brutes, en particulier des fibres usinées à sec; ce problème est largement atténué par un défibrage préalable modéré;
  - l'analyseur FS-100 surestime quelque peu les populations de fibres courtes. Ceci peut être expliqué soit par le comptage occasionnel de particules non fibreuses, soit parce que le capteur optique de l'instrument ne détecte qu'une partie des fibres longues qui seraient trop minces, courbées, partiellement ouvertes, etc.;
  - la longueur moyenne des fibres (en population ou normalisées) calculée par le Kajaani FS-100 est plus faible que celle prédite selon les formules de calcul données dans la littérature Kajaani (ceci semble un problème de logiciel plutôt qu'un problème du système de mesure);
  - puisque l'analyseur Kajaani ne donne pas d'information sur le diamètre des fibres, l'évaluation quantitative des fractions de poids dans chaque classe de longueur requiert une connaissance additionnelle, ou des hypothèses de travail, sur les diamètres de fibres.
- Le Kajaani FS-100 semble idéal pour caractériser des fibres ayant des longueurs correspondant aux fractions Bauer-McNett entre +200-mailles et -4-mailles (le Kajaani FS-100 ne détecte pratiquement aucune fibre dans la classe de longueurs correspondant à la fraction +4-mailles).
- Un usage routinier de l'analyseur FS-100 pour fins de contrôle de qualité ou surveillance de procédés nécessitera donc une mesure

indépendante des fractions -200-mailles et +4-mailles (voir ci-dessous); l'utilisation du Kajaani serait donc préférée dans le cas de fibres produites en voie humide et ayant de faibles teneurs en poussières.

8 - COMPARAISON DES DONNÉES DE LA CLASSIFICATION BAUER-McNETT AVEC LES ANALYSES DIMENSIONNELLES PAR MICROSCOPIE OPTIQUE ET À L'AIDE DU KAJAANI FS-100

L'ensemble des données sur les longueurs et diamètres de fibres, obtenues par microscopie optique ou avec l'analyseur Kajaani, constitue une excellente base de données pour définir les paramètres dimensionnels «réels» des fibres à l'intérieur des différentes fractions Bauer-McNett. Une analyse détaillée des résultats, comparant les données Bauer-McNett et les paramètres dimensionnels mesurés directement, mène aux conclusions suivantes:

- tel qu'anticipé, la longueur moyenne des fibres obtenues dans les diverses fractions Bauer-McNett ne correspond pas à la dimension des ouvertures des tamis;

- la fraction -200-mailles de la classification BMN contient des quantités substantielles de fibres ayant des longueurs plus grandes que 100 µm (l'ouverture d'un tamis 200 correspond à 70 µm); ceci devient particulièrement évident avec le groupe «S» des fibres humides de Cassiar;

- la fraction BMN +4-mailles qui correspond au groupe des fibres les plus longues contient une quantité substantielle de fibres relativement courtes, présumément en raison de la formation d'agrégats (par adhésion ou accrochage des fibres dans la suspension aqueuse), (les agrégats formés ne passant pas le tamis 4-mailles);

- les données obtenues par microscopie optique ont été utilisées pour calculer les «ouvertures effectives» de tamis qui mèneraient à un accord entre les distributions en poids obtenues par microscopie optique et celles obtenues par la classification BMN; cette approche ne s'est pas avérée pratique puisque les ouvertures effectives se sont révélées dépendantes du type de fibre, ce qui était assez prévisible;

- Par ailleurs, ayant noté que le diamètre moyen des fibres défibrées au Pallmann est relativement constant (varie peu selon la longueur des fibres), nous avons utilisé les distributions de longueurs obtenues au Kajaani pour reconstituer les classifications BMN en supposant:

    - un diamètre moyen unique pour toutes les fibres (la valeur précise n'a pas d'importance);

    - des classes de longueurs de fibres dont les points de coupure correspondent à la taille des ouvertures des tamis BMN;

    - l'absence de la fraction -200-mailles.

  Les graphiques BMN ainsi reconstitués à partir de distributions de longueurs sont comparés aux graphiques BMN expérimentaux dans les Figures 4 et 5, respectivement pour les fibres humides et sèches.

- L'accord entre les fractions BMN mesurées et calculées ne peut être quantitatif puisque nous avons ignoré la fraction -200-mailles dans les calculs; la comparaison est néanmoins intéressante puisqu'elle permet de constater que, dans une fraction BMN donnée, l'ordre relatif prédit pour les divers types de fibres est adéquatement prédit.

  Puisque les graphiques BMN «reconstitués» ont été calculés à partir des distributions Kajaani normalisées il serait évidemment beaucoup plus pertinent d'utiliser directement ces distributions de longuers pour fins de comparaison ou de contrôle de qualité.

FIBRES CASSIAR, VOIE HUMIDE

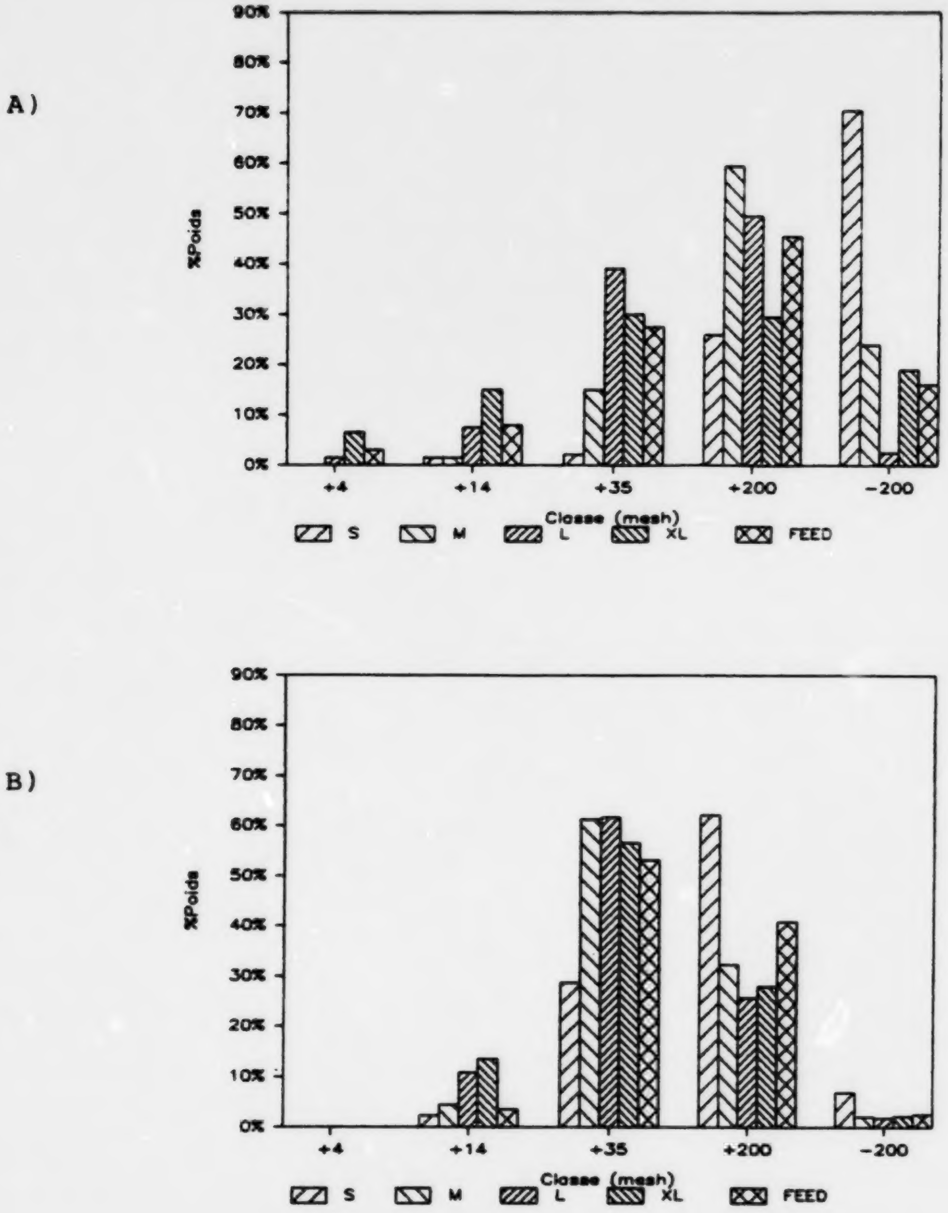

FIG. 4 A) Classifications Bauer-McNett, résultats expérimentaux

B) Classifications Bauer-McNett simulées, reconstituées à partir des distributions de longueurs obtenues au Kajaani

FIBRES CASSIAR, VOIE SÈCHE

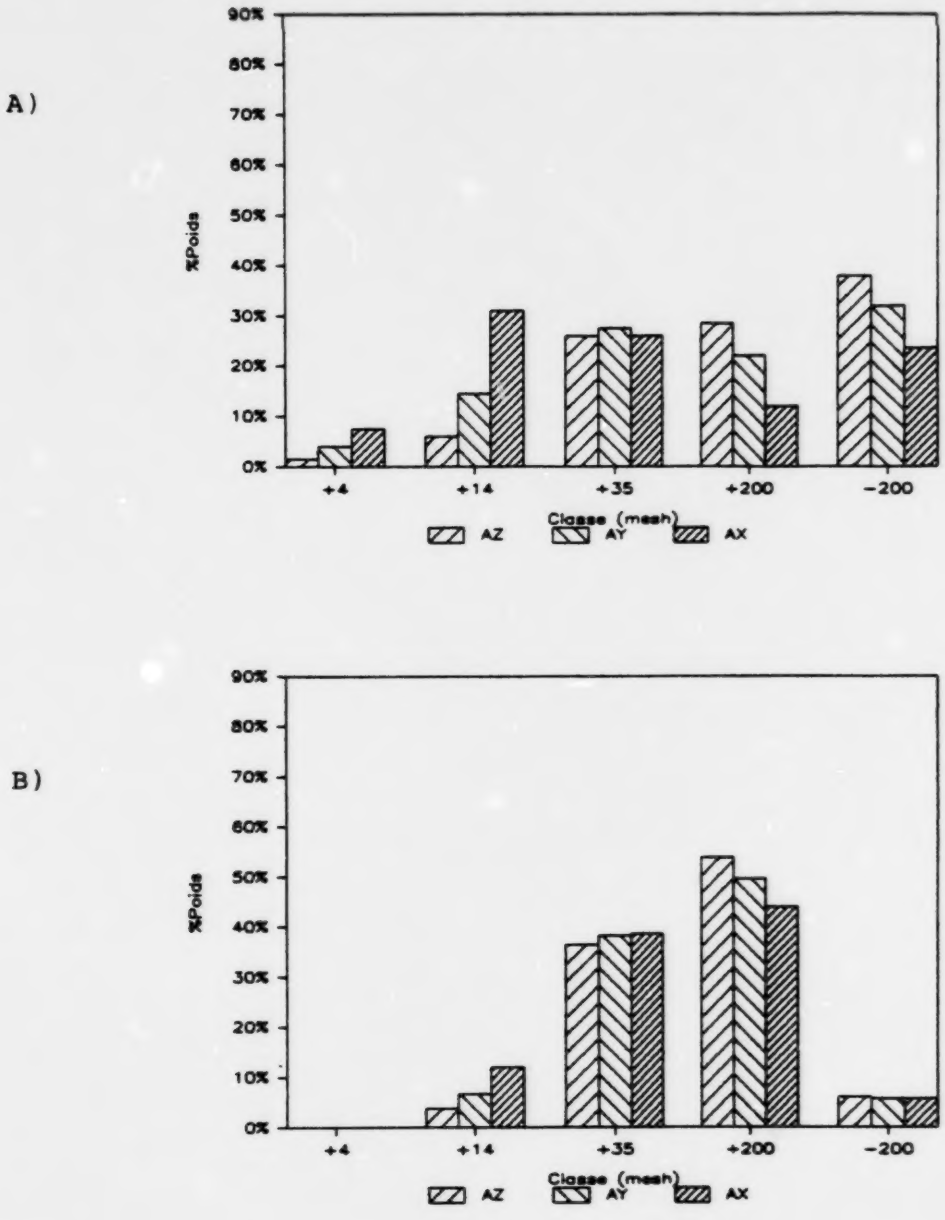

FIG. 5 A) Classifications Bauer-McNett, résultats expérimentaux

B) Classifications Bauer-McNett simulées, reconstituées à partir des distributions de longueurs obtenues au Kajaani

9 - ESSAIS DE FORCE TENSILE DANS LES COMPOSÉS D'A/C (FSU)

Les essais de force tensile[1] dans l'amiante-ciment ont été effectués pour les divers types de fibres étudiées après défibrage au Pallmann à 6500 rpm. Le défibreur Pallmann s'est avéré adéquat pour les fibres Cassiar produites en voie humide; toutefois, nous avons démontré par la suite que les fibres Cassiar produites en voie sèche (AZ, AY et AX) nécessitaient un défibrage plus intense afin d'obtenir des valeurs optimales en FSU. Les principaux résultats de FSU sont rassemblés au Tableau 5.

TABLEAU 5: Résultats des essais de force tensile (FSU) sur fibres Cassiar

	Méthode d'ouverture	Densité lâche (lb/pi^3)	% poids de fibre dans mélange A/C	Module de rupture ajusté (kg/cm^2)	FSU
Extraction sèche					
AZ	Pallmann	4.4	16.3	255.9	57.8
AY	"	2.7	11.4	261.7	84.0
AX	"	1.9	8.8	256.4	106.6
AZ	Broy. boulets & BOP		12.5	294.9	85.1
Extraction humide					
S	Pallmann	0.7	4.3	186.8	---
S	"		6.0	188.7	---
M	"	1.3	6.8	251.7	135.4
L	"	1.5	7.6	262.8	126.2
XL	"	1.3	6.8	256.7	138.0
FEED	"		7.5	275.0	133.0
S	Turbine		6.2	202.6	---
M	"		7.4	278.5	136.7
L	"		7.9	254.4	117.8
XL	"		7.2	274.9	138.8

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[1] Les essais ont été effectués à Ceram-sna, Sherbrooke.

Les résultats FSU soutiennent les conclusions suivantes:

- Sous des conditions identiques de défibrage, les fibres extraites en voie humide mènent à des valeurs de FSU plus élevées que les fibres de longueurs comparables produites par voie sèche; ceci est vraisemblablement dû à un degré de défibrage plus élevé pour les fibres humides: suite au traitement Pallmann, le diamètre moyen des fibres humides est environ 50% inférieur à celui des fibres sèches.

- Toutes les fibres humides étudiées, à l'exception du groupe «S», affichent d'excellentes propriétés de renfoncement (FSU entre 120 et 140) lorsque défibrées au Pallmann (à sec) ou à la turbine (humide).

- Les fibres de type «S» confèrent au ciment un renforcement flexural substantiel même à faible teneur en fibres; cependant, le module de rupture demeure faible (180 à 200) même si la teneur en fibres est augmenté; ceci empêche le calcul d'une valeur utile de FSU.

- Les fibres usinées et classifiées par voie humide (M, L, XL) mènent à des valeurs semblables de FSU; ceci est en accord avec le fait que les fractions M, L et XL ont des longueurs et des diamètres comparables.

- Les valeurs de FSU obtenues pour les fibres humides brutes (FEED) sont comparables à celles des fractions M, L et XL, suggérant que seule une classification minimale serait requise pour applications dans les produits d'amiante-ciment.

- Dans le cas des fractions S, M, L et XL, le défibrage en milieu aqueux à l'aide d'une turbine mène à des valeurs de FSU comparables à celles obtenues par défibrage au Pallmann (après séchage).

10 - MÉTHODES ALTERNATIVES DE DÉFIBRAGE

Dans la majorité des essais effectués dans la présente étude, nous avons utilisé le défibreur Pallmann et l'influence de ce mode de défibrage sur les fibres a été étudié en détail. Par ailleurs, d'autres modes de défibrage ont également été examinés, ce qui a mené aux conclusions générales résumées ci-dessous.

10.1 - Défibrage Pallmann

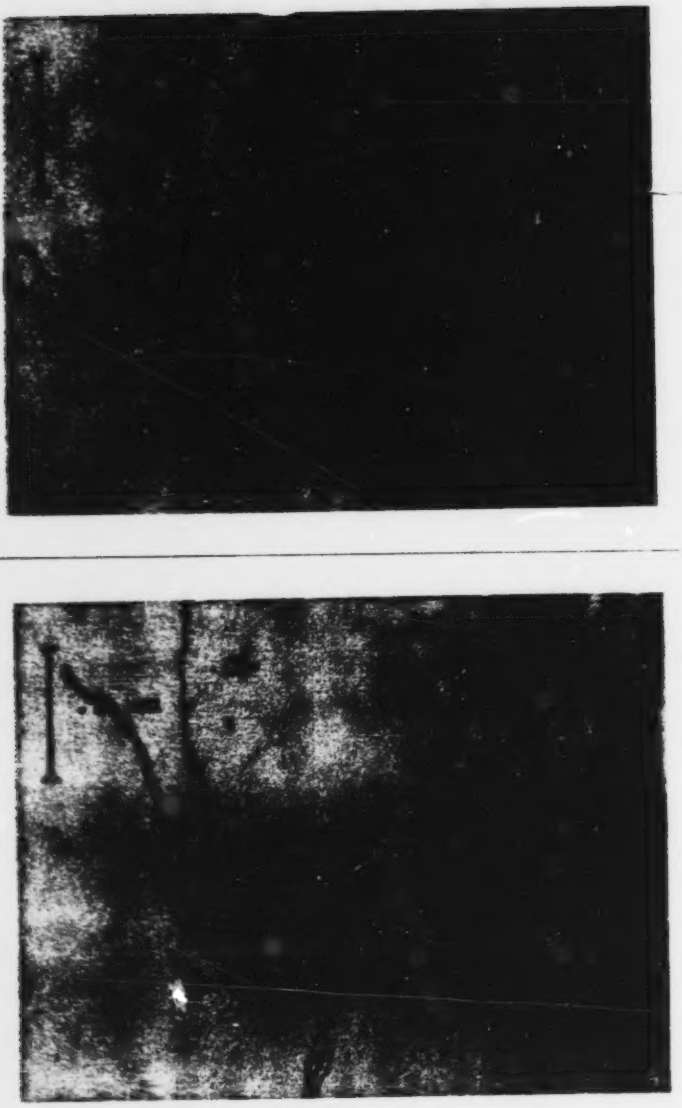
Nous avons observé que le défibrage de fibres brutes à l'aide du défibreur Pallmann à 6500 rpm conduit à une légère réduction des longueurs moyennes des fibres (∿ 10%); une réduction des diamètres moyens fut également atteint, réduction qui s'avère marquée surtout pour les fibres ayant des diamètres supérieurs à 0,01 mm (ou 10 µm). Cette observation, couplée aux informations recueillies à l'examen microscopique des fibres, avant et après traitement au Pallmann, nous a conduit aux conclusions suivantes:

- sous les conditions de la présente étude, le défibreur Pallmann réduit peu le diamètre des fibres lorsque celles-ci ont un diamètre inférieur à 10 µm;

- les fibres passées au défibreur Pallmann montrent fréquemment des écrasements, des cassures et une ouverture partielle tel qu'illustré à la Figure 6. La présence de telles déformations contribue à réduire leur densité lâche et tend à augmenter leur diamètre moyen apparent; ces déformations jouent vraisemblablement un rôle important dans le renforcement du ciment (c'est-à-dire offrant des points d'ancrage additionnels pour les particules de ciment);

- pour les fibres Cassiar usinées à sec, un seul passage au défibreur Pallmann ne conduit pas à une ouverture optimale pour le renforcement du ciment.

FIBRES CASSIAR



a) 6500 rpm

b) 13000 rpm

FIG. 6 Fibres humides fraction "S" de Cassiar traitées au Pallmann à deux vitesses rotatives (agrandissement: 100X; barre: .2mm)

10.2 - Défibrage à la turbine

L'agitation vigoureuse prolongée d'une suspension aqueuse de fibres dans un appareil de type turbine (un contenant avec des chicanes et un agitateur) mène à un défibrage substantiel, sans, toutefois, réduire de façon significative la longueur moyenne des fibres. Dans ce type d'appareil, l'effort transversal exercé sur les fibres est plus faible que dans un défibreur Pallmann, de sorte qu'on observe peu de déformation du type écrasement ou cassure.

10.3 - Défibrage au tube de Dounce

Le défibrage résultant du passage forcé d'une suspension aqueuse de fibres entre la paroi d'un cylindre et un piston de forme sphérique mène à des fibres très ouvertes. Le principe exploité dans le tube de Dounce est le même que celui utilisé dans le broyeur Szego. Une comparaison détaillée des longueurs et diamètres moyens révèle une réduction des diamètres plus importante que celle obtenue par défibrage au Pallmann, bien que l'on observe pas de réduction significative des longueurs moyennes.

Le tube de Dounce est une méthode de faible échelle qui semblerait idéale pour un défibrage préalable à la mesure des distributions de longueurs avec l'analyseur Kajaani.

10.4 - Méthodes ultrasonores

Une irradiation ultrasonore prolongée d'une suspension aqueuse de fibres mène simultanément à un défibrage et à une rupture des fibres. En fait, l'irradiation prolongée à haute puissance conduit à de très fines fibres ayant des longueurs moyennes de l'ordre de 10 µm!

11 - MÉTHODES DE MESURE DE L'AIRE SPÉCIFIQUE

En rapport avec l'objectif d'évaluation quantitative du degré de défibrage, nous avons exploré trois (3) méthodes susceptibles de nous fournir des renseignements sur l'aire spécifique et la porosité des fibres:

- l'aire spécifique BET à l'azote
- la porosité au mercure
- l'adsorption de colorants en solution.

Bien que notre étude fut limitée à quelques fibres (humides et sèches, brutes et défibrées), certains résultats intéressants ont été obtenus, lesquels indiquent des directions prometteuses pour des travaux ultérieurs:

- Les valeurs d'aires spécifiques BET mesurées à l'azote affichent des tendances semblables aux valeurs d'aires de surface rapide (RSA); toutefois, la contribution de la fraction des poussières (c.-à-d. -200-mailles) semble beaucoup plus importante pour l'aire spécifique BET que pour les valeurs de RSA; ceci suggère une approche potentielle pour évaluer quantitativement la teneur en poussières (fraction -200-mailles) d'un échantillon.

- Les résultats de porosité au mercure affichent des différences marquées entre les fibres brutes et les fibres traitées au Pallmann (environ 90% du volume des pores se retrouve avec un rayon variant entre 0.1 et 3 µm); on observe également de faibles différences entre le comportement des fibres extraites en voies humide ou à sec. Cette méthode apparaît bien adaptée pour évaluer quantitativement le degré de défibrage.

- L'étude de l'adsorption d'un colorant organique ("Acid Black") sur des fibres en suspension aqueuse conduit à deux types d'informations quantitatives reliées respectivement à l'aire spécifique et à l'affinité des molécules organiques pour le solide. Nos résultats montrent de façon concluante que l'aire spécifique peut être obtenue à partir des mesures d'adsorption de colorants directement sur les fibres humides; toutefois, la relation entre les paramètres d'adsorption et l'aire spécifique dépend du type de fibre (c.-à-d. des fibres ayant des affinités différentes); ainsi, la méthode nécessitera un «étalonnage» pour chacun des types de fibres étudiés.

12 - ORIENTATION DE RECHERCHES ULTÉRIEURES

En relation avec les objectifs à long terme de ce travail, notamment, le développement de méthodes automatisées pour la caractérisation physico-chimique de fibres industrielles de chrysotile, les données obtenues de la présente étude suggèrent les étapes stratégiques suivantes (en série ou en parallèle):

- Effectuer une étude similaire sur une variété plus étendue de fibres de chrysotile extraites à sec, d'abord pour des fibres d'origine canadienne, et ensuite quelques fibres de compétiteurs.

- Poursuivre une étude quantitative de l'adsorption de colorants par des fibres en suspension aqueuse afin de mettre au point une méthode de mesure d'aire spécifique directement en suspension.

- Examiner diverses approches pour mesurer de façon automatique la teneur en poussières et particules non fibreuses; pour des fibres extraites en procédé sec, il semblerait utile d'exploiter les différences entre les valeurs de l'aire de surface rapide (RSA) et les valeurs d'aire spécifique BET; pour des fibres humides, la différence entre l'affinité des fibres et celle des particules non fibreuses vis-à-vis les colorants organiques pourrait être avantageusement exploitée.

- Étudier les méthodes pour évaluer in situ la teneur en fibres d'une suspension aqueuse dans un circuit d'usinage.

- Examiner plus à fond les relations entre la porosité au mercure et le degré de défibrage des fibres (extraites à sec, ou séchées après extraction humide).

- Poursuivre des travaux sur l'utilisation des microondes comme source de chauffage, soit pour fins de séchage de fibres humides, soit comme pré-traitement au défibrage mécanique.

- Étudier la performance de divers équipements de type turbine pour le défibrage humide en vue d'incorporation éventuelle dans un circuit d'extraction en voie humide.

- Poursuivre des travaux sur les méthodes ultrasonores de défibrage en suspension aqueuse (possiblement assisté par des dispersants dont l'effet ne serait pas détrimentaire pour les produits d'amiante-ciment).

- Réaliser une étude plus détaillée des conséquences de l'extraction en voie humide sur la teneur de divers contaminants retrouvés dans les fibres chrysotile (c.-à-d. oxyde de fer, magnétite, carbonate, brucite, némalite, etc.).

- Conjointement avec les études mentionnées ci-dessus, poursuivre des travaux à l'échelle pilote pour mettre au point une stratégie détaillée de surveillance et de contrôle automatisé.

RECOMMENDATIONS

With regards to specific issues on wet-milling processes and products, the results of the present study substantiate the following recommendations:

1 - Wet-milling processes can be favorably envisaged to produce very "clean" fibers which are comparatively easy to open and which perform very well in asbestos-cement products.

2 - The wet classification of the bulk wet-milled fiber products (FEED) into four subgroups (S, M, L and XL) leads to a mild opening of the fibers. Although the properties of the S fibers are different from those of the M, L and XL fibers, the properties of the latter three were found closely similar. Hence, if the S fraction is not detrimental to the envisaged applications, extensive classification procedures would not be required.

3 - For asbestos-cement applications, it would seem advantageous to include a turbine-type opening step in the wet-milling circuit. Proper wet opening of the bulk wet-milled fibers (FEED) would yield a group of fibers which could be incorporated directly into cement slurries.

4 - A fiber length analyser, the Kajaani FS-100 or an equivalent instrument, could be used advantageously to complement or replace BMN classification tests. The Kajaani FS-100 Fiber Length Analyser can rapidly yield precise fiber length distributions which may be used for fiber quality control, or in milling circuit monitoring; the following restrictions, must, however be acknowledged:

  - to relate the fiber length distributions to weight fractions by length groups, the fiber sample must be opened to reduce the average diameter to a constant (reproducible value); Pallmann or Dounce tube opening appeared adequate;

- ensuring constant average diameters, the normalized fiber length distributions may be used for comparisons or monitoring purposes; they relate most directly to weight fractions;

- grit particles and coarse fiber bundles must be prevented from entering the measurement circuit;

- the dust fraction normally found within the -200-mesh BMN fraction must be measured independently (e.g., elutriation);

- the Kajaani FS-100 tends to overestimate the short fiber groups, but this should not be a major drawback;

- samples of reference fibers should be provided or developed to allow occasional verification of instrument calibration.

5 - To eventually replace the BMN test with a Fiber Length Analyser, an extented period of parallel measurements should be carried out for statistical validation and user acceptance of the results.

6 - The surface area of wet-milled fibers can be determined in aqueous slurries with dye adsorption methods. This is not as straightforward as expected, mainly because the amount of adsorbed dye depends both on the surface area and on the type of fiber.

7 - Dye adsorption and other methods explored in this project should be investigated further to distinguish quantitatively, and directly in aqueous slurries, the relative amount of dust and fibers.

8 - Other specific recommendations for follow-up work in relation to automated monitoring of fiber milling and fiber quality control are outlined in the "Executive Summary" (Section 12).

I - INTRODUCTION

The Canadian asbestos industry produces a broad variety of commercial fibers which are designated in several main groups 3, 4, 5, 6, 7 or equivalent, each comprising numerous sub-groups specific to individual producers. The characterization of these fiber products according to their intrinsic properties (e.g., length, diameter, openness, dust, grit, magnetite contents, etc) and to user-relevant criteria (e.g., cement reinforcement and drainage properties) has always been a prime concern of the asbestos industry. The Canadian asbestos industry, in particular through the Quebec Asbestos Mining Association, has played a leading role in developing standard tests for fiber quality control.

Traditionally, the asbestos production industry has relied on partial characterization of certain properties deemed most relevant to the user industries. For this purpose, a series of practical testing procedures were developed which, in the case of chrysotile asbestos, are described in the "Standard Chrysotile Tests" manual[1]. These tests were developed by the Quebec Asbestos Mining Association, Turner and Newall, the Asbestos Textile Institute and others and comprise more than thirty testing protocols.

Typically, the standard tests define some dimensional features of the fibers (e.g., Bauer-McNett fiber classification), their cleanness (e.g., Turner and Newall dust elutriation) or their behavior in specific processes and products (e.g., "freeness" test for drainage rates in asbestos-cement (A/C) processes: "fiber strength unit" test for reinforcement in A/C products).

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[1] Asbestos Textiles Institutes, Inc., "Chrysotile Asbestos Test Manual". 3rd ed. 1974, rev. 1978, Hatboro, PA, USA, 1978, 1 v.

These tests fill an essential function in providing fiber quality control and quality assurance methods for the producer and user industries, using relatively small fiber samples and, usually, with instruments of minimal sophistication. The standard tests, however, provide little direct information on the basic properties which are responsible for the behavior of chrysotile fibers in industrial processes, and their performance in asbestos containing products.

In recent years, several factors have stressed the importance of optimal fiber characterization procedures:

- First, increasing competition from foreign asbestos producers raised the need for detailed quantitative comparisons of fiber properties. The same is also critically important with regards to substitute fibers which are usually supplied with exhaustive characterization and technical specifications.

- Second, the potential improvement of fiber quality in existing dry-milling installations (e.g., through equipement development, process modifications, computer assisted process monitoring and control) will require rapid methods to assess the relevant properties of the fiber products.

- Third, the introduction of new asbestos milling technologies, for example wet milling processes, yields new fiber products, the properties of which need to be adequately compared to dry-milled fibers.

All of these situations point to a common need for the developement of fiber characterization methods which would apply to all types of chrysotile fibers and which would take full advantage of modern physicochemical techniques. In response to this need, we have elaborated a systematic research program which, if pursued to completion should provide the following:

- An understanding of the relationship between the results of "Standard Chrysotile Tests" and several dimensional and morphological parameters of the fibers;
- The development of alternative fiber assessment methods in relation to their required end-use properties (behavior in processes, properties in products);
- The identification of key fiber parameters which could serve as basis for the development of automated fiber testing methods;
- The design of strategies for the automation of fiber quality control in milling operations.

The results described in the remainder of this report relate to the first of these general goals and partly to the second, in the particular case of wet- and dry-milled fibers produced by Cassiar Mining Corp. Similar work pertaining to other types of Canadian chrysotile fibers, as well as the other issues identified above, will be addressed in later research projects.

II. SPECIFIC OBJECTIVES AND SCOPE OF WORK.

Within the general research goals outlined above, the specific primary objectives of the work reported here were to:

- Provide detailed dimensional characterization of several chrysotile fiber groups produced by Cassiar Mining Corp. through wet- and dry-milling technologies, using all relevant physico-chemical and size determination methods.

- Establish qualitative and quantitative relationships between the results of standard chrysotile tests and the true dimensional (or other physico-chemical) properties of the fibers.

- Provide basic information and data for later developments of alternative fiber testing methods and on-line monitoring of asbestos milling processes.

Additional issues specifically related to the Cassiar wet-milling process were raised at the onset of the project, or during the course of the work; these can be summarized as follows:

- Establish the effectiveness of the Cassiar wet fiber classification technologies.

- Provide an assessment of a fiber-length analyzer (marketed by Kajaani) for the measurement on chrysotile fiber length distribution.

- Carry out laboratory testing of alternative wet-fiber opening methods (ultrasounds, water turbines or other) and provide a characterization of the fiber products.

- Suggest practical strategies for on-line monitoring of fiber length distributions in wet-milling circuit.

- Investigate dye-adsorption methods for evaluating the surface area (openness) of fibers in aqueous slurries.

- Carry out a preliminary examination of various other types of Canadian dry-milled fibers using the Kajaani (FS-100) Fiber Length Analyzer.

A detailed strategy to achieve the primary objectives is outlined in Section IV, following a brief review of the fiber characterization methods currently available (standard chrysotile testing methods and complementary methods) in Section III.

III - OVERVIEW OF METHODS FOR FIBER CHARACTERIZATION: "STANDARD CHRYSOTILE TEST" AND DIRECT MEASUREMENT METHODS

III.1 - Characterization of chrysotile fibers: General requirements and traditional approaches

The "full" characterization of a fibrous material requires data on each of the following dimensional and physico-chemical aspects:

- the distribution of lengths (ℓ), diameters (d) and aspect ratios (ℓ/d) of the fibers;
- the chemical composition and structure of the fibers;
- the surface properties (surface charge or potential, surface area and porosity, adsorption behavior, etc.);
- other physical properties: mechanical, electrical, magnetic, etc.

In the case of industrial grades of chrysotile fibers, the "full" characterization will require additional knowledge, typically:

- extent of partial "opening" (each fiber consists of a bundle of microfibers which may be partially opened);
- the presence of other fibrous materials, typically nemalite and tremolite;
- dust, grit and spicule contents;
- magnetite, brucite, carbonate, etc. contents.

As mentioned earlier, the asbestos production industry has traditionally relied on partial characterization of certain properties deemed most relevant to the user industries. The set of "Standard Chrysotile Tests" developed to provide the user industries with operational criteria comprises more than thirty (30) testing protocols grouped according to:

Dry fiber length (5 tests).
Wet fiber length (5 tests).

Openness, loftiness and drainage (7 tests).
Crude and granular content (2 tests).
Strength (3 tests).
Other physico-chemical properties (10 tests).

III.2 - Dimensional characterization of chrysotile fibers: Standard methods and proposed direct measurement methods

We outline below the basis of a comprehensive dimensional characterization of chrysotile fibers according to:

- standard testing methods and user relevant properties;
- other methods applicable for fiber dimensional analysis.

As most (70%) of the chrysotile asbestos currently used is in A/C products, the standard tests considered are those most relevant to this particular industry.

III.2.a - Standard methods and user relevant properties

The series of standard chrysotile tests upon which the A/C industry relies most for fiber acceptance and quality control purposes are listed below. For each of these, we indicate:

- the conditions of applicability;
- the significance of the results obtained;
- the known or expected relationship between the measured properties and the dimensional parameters of the fibers.

a - Ro-Tap screen analysis

Applicability : Dry sieving method used for short fibers (Group 4 and shorter).

Significance : Provides a separation of fiber into several fractions of decreasing "average size".

Related size parameters : Apparent fiber size, presumably weighted by the aspect ratio of the fibers, i.e. the probability that a fiber will pass through a hole the size of its diameter will decrease as its aspect ratio increases; at $\ell/d = 1$, this probability will be unity.

b - Elutriation analysis

Applicability : All commercial fiber grades, preferably the longer groups using air or water as suspending medium.

Significance : Provides a quantitative estimate of the fines (very short fibers, dust particles) in the commercial fibers, i.e., cleanness.

Related size parameters : Elutriation separates according to hydrodynamic radii (r_h). Air elutriation is quoted to remove particles less than 40 μm diameter; in water elutriation, the cut-off upper r_h value does not appear to have been reported.

c - Bauer-McNett wet classification

Applicability : Wet sieving method widely used for all commercial grades (preferably for groups 3 or shorter fibers; long fibers yield more erratic results).

Significance : Provides separation of commercial grades into several standard sub-groups using 4/14/35/200-mesh screens.

Related size parameters : Average fiber size dependent on the hydrodynamic radii (r_h) of the fibers in water an on their aspect ratio (see "Ro-Tap analysis" above).

d - Dyckerhoff air permeability and Rapid surface area

Applicability : Dry-milled commercial fiber grades having Dyckerhoff time of efflux from 10 to 600 sec. and rapid surface areas in the range 0,1 to 2,5 m^2/g.

Significance : Provides a measure of macroscopic porosity of a bulk dry fiber sample.

Related size parameters : Air permeability is related to the degree of opening of dry-milled chrysotile and to its surface area.

e - Freeness

Applicability : All commercial grades in groups 4-7.

Significance : Provides quantitative values for rates of dewatering of chrysotile fibers in water saturated with lime and gypsum.

Related size parameters : Degree of openness, surface area and weight fraction of fines (very short fibers and dust); freeness is also very likely to depend on the chemical composition of the fibers.

f - Fiber strength in A/C (Fiber Strength Unit, FSU)

Applicability : All commercial fibers preferably those of groups 4 to 6.

Significance : Measures directly one of the key properties of A/C compositions: the flexural strength imparted by chrysotile fibers to cement.

Related size parameters : Average length, average diameter, aspect ratio degree of opening and surface area; fiber strength in A/C may also be related to surface chemical properties of the fibers.

It is noteworthy that the results of the various testing methods listed above all depend on fiber size and morphology; however, none of the results are uniquely related to one, or a few, well defined size parameters.

III.2.b - Alternative methods applicable to fiber size characterization

We list below several size characterization methods which can be used to obtain dimensional properties of fibers in terms of averages or distributions of the measured parameters: ℓ , d, ℓ /d, surface area. For each property, we indicate the applicability (confirmed or expected) of the method and the size parameters which can be derived.

a - Optical microscopy (OM)

Applicability : Fiber having lengths greater than 5 µm; the resolution limit of a good optical microscope is of the order of 0,2 µm.

Parameters obtained : Distributions of fiber lengths, diameters and aspect ratios.

b - Transmission electron microscopy (TEM)

Applicability : Fibers longer than 0,1 µm but preferably shorter than 100 µm (resolution limit of 0,01 µm): most useful for shortest fibers.

Parameters obtained : Same as OM.

c - Kajaani fiber length analyser

Applicability : Dilute suspensions of fibers longer than 50 µm and with minimal unspecified diameter (resolution limit quoted as 50 µm).

Parameters obtained : Length distribution.

d - Light scattering and electrophoretic mobility

Applicability : Dilute aqueous suspensions of fibers; best applies to short fibers, e.g., < 100 µm.

Parameters obtained : Distribution of hydrodynamic radii in water and distribution of fiber electrophoretic mobilities which depend on surface charge.

e - Gas adsorption surface area (N_2)

Applicability : Dry, degassed fibers of any size group.

Parameters obtained : Surface area, mainly reflecting fiber openness (i.e. average diameter and micro-porosity).

f - Dye adsorption surface area (method to develop)

Applicability : Aqueous fiber slurries.

Parameters expected : Similar to N_2 surface area, reflecting fiber openness , micro-porosity, dust/fiber ratio.

IV - RESEARCH STRATEGY

IV.1 - Choice of fiber types

An important long range goal of the present work is to enable the prediction of chrysotile fiber performance in a given product, based upon dimensional, morphological and other physico-chemical parameters of the fibers. At the onset, a trivial, but most important point must be emphasized: in most applications, and particularly in asbestos-cement (A/C) products, the bulk commercial fibers are subject to extensive "opening" in the user process. Hence, the various tests performed on the bulk commercial fibers can have only limited value for predicting fiber performance in a given process or product. Consequently, to meet with requirements from both the producer and the user industries, we need to pursue detailed dimensional characterization of the industrial fibers, first as provided, and second, following extensive opening as carried out, for example, in the FSU testing protocol[1].

To provide relevant data on the various issues of the present investigation, the materials selected must therefore comprise:

- wet-milled and dry-milled fibers;
- bulk wet-milled fiber products and wet-classified fiber groups;
- industrial fibers and "opened" industrial fibers.

Finally, because various factors other than size and morphology are likely, and in some cases known, to influence the behavior and properties of chrysotile fibers (e.g. the iron oxides, brucite, carbonate, chloride, etc. contents; the intrinsic micro-structure; elemental lattice substitution), a "full" characterization of these fibers would require considerably more than the examination of size-related parameters. However, as we are mostly dealing here with a set of wet- and dry-milled fibers of the same origin, we may assume that the main chemical and microstructural factors remain constant.

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[1] Chrysotile Asbestos Test Manual, Section F-2.

IV.2 - Basis for dimensional analysis and correlation of properties

The present study draws upon a wide variety of methods which yield parameters related to fiber size and morphology. To help develop a comprehensive research plan, we devised a classification of these methods. The latter distinguishes four levels of analytical or testing approaches according to the type of dimensional information they provide; the criteria used are summarized below:

Level	Criteria
Primary	Methods which enable direct measurement of fiber size parameters (ℓ, d).
Secondary	Properties which yield direct information on size parameters other than ℓ or d, for example the surface area.
Tertiary	Methods yielding results which depend simultaneously on several size parameters for example, the Bauer-McNett test.
Quaternary	Methods yielding results which are related to size parameters, but to other properties as well, for example, the elutriation test.

The classification of methods within this hierarchy is illustrated in Table IV.1. The appropriate overall research strategy is then readily seen as follows:

- the primary and secondary methods are used to obtain a full dimensional characterization of the fibers;
- the parameters provided through studies using ternary and quaternary methods are then correlated with fiber size and morphology parameters (obtained from primary and secondary methods);
- the correlations establish the relative importance of the various parameters for user-relevant properties and the significance of standard test results;
- the same correlations will also point to any differences between dry- and wet-milled fibers, differences in fiber opening methods, etc.

The detailed research plan and experimental protocols were designed within this overall strategy.

TABLE 1: Classification of fiber characterization methods

LEVEL	**METHODS**		
PRIMARY	OPTICAL MICROSCOPY $(\ell ; d)$[1]	TRANSMISSION ELECTRON MICROSCOPY $(\ell ; d)$	KAJAANI (ℓ)
SECONDARY	LIGHT SCATTERING (r_h)	NITROGEN SURFACE AREA (S.A.; d)	DYE ADSORPTION IN SOLUTION (S.A.; d)
TERTIARY	BAUER-McNETT $[\ell , r_h]$[2]	AIR PERMEABILITY [S.A.]	FIBER STRENGTH UNIT $[\ell ; d;$ S.A.]
QUATERNARY	FREENESS [S.A.]	ELUTRIATION $[r_h]$	

1 - Entries in parenthesis indicate the size parameters measured.
2 - Entries in brackets indicate size parameters upon which the results are believed to depend.
(S.A.: surface area; r_h: hydrodynamic radii)

V - METHODOLOGIES

As noted earlier, the present study called upon a variety of standard chrysotile testing methods and other physico-chemical techniques to obtain dimensional information or other relevant data. The standard tests performed comprised: Bauer-McNett, T&N elutriation, mag-rating, A/C fiber strength, bulk density and rapid surface area. These tests were performed in the quality control laboratories at Cassiar Mining Corp., Cassiar, B.C., except for the fiber strength tests which were carried out at Ceram-sna inc., Sherbrooke.

For the standard chrysotile tests, the established protocols[1] were followed in all cases except for one particular aspect of the fiber strength test: a Pallmann mill was used to (open) defiberize the crude fiber samples instead of a ball-mill/BOP combination recommended in the standard protocol. This substitution was made on the following grounds:

- the Pallmann opening mill exerts less impact and transverse shear action than the combined ball-mill/BOP procedure;

- the Pallmann was shown effective in adequately opening the various fiber types from Quebec asbestos producers.

Experiments with alternative fiber opening devices were also carried out as described in Section VII.3. The physico-chemical methods used in the characterization of the various fiber types studied here are listed in Table V.1. For each case, we report the property examined, the type of equipement and the type of information extracted from the results. Brief comments on the methodologies and on the significance of the results are given below.

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[1] Detailed description of these tests and their exact procedures are available at ASTM publications, and in the CHRYSOTILE TESTS MANUAL, released by QAMA (Quebec Asbestos Mining Association).

TABLE V.1: Physico-chemical techniques used in this work, in addition to standard chrysotile testing methods(1)

Technique	Types of instrument	Information/data
Nitrogen Surface Area (SA)	Quantachrome	Surface area accessible to a nitrogen molecule (in m^2/g)
Infrared Spectroscopy (IR)	Nicolet MX-1	Identification of functional groups and chemical bonds
Thermogravimetric Analysis (TGA)	Perkin-Elmer TGS-2	Quantitative analysis of some chrysotile contaminants (e.g., brucite)
Optical Microscopy Photography (OM)	Leitz Laborlux 12 (microscope) Wild MPS51 (camera) Wild MPS45 (photometer)	High quality microscopic frames of fiber samples
Length and Diameter Sampling (from OM frames)	HIPAD digitizer Bioquant II (soft ware)	Distributions of fiber lengths and diameters
Scanning Electron(2) Microscopy (SEM)	JEOL JSM-25 SIII Scanning microscope	Same as OM, at higher magnification and resolution
Fiber Length Analysis	Kajaani FS-100	Length distributions
Mobility in electric field	ZETASIZER IIc	Apparent surface charge
Dye adsorption	UV-Visible	Surface area accessible to large organic molecules in aqueous fiber slurries
Mercury porosimetry	Carlo Erba 200 (150 MPa)	Pore-size distribution

(1) See following pages for details of the methods.
(2) SEM was used rather than TEM for reasons given in Section V.6.

V.1 - Nitrogen surface area

The nitrogen surface area mesurement is based on the ability of gas molecules to be adsorbed at a gas/solid interface. By measuring the gas desorption volume from a sample which was submersed in liquid nitrogen, and using the BET type II isotherm model, the surface area accessible to the gas molecule can be evaluated. Since nitrogen molecules have a radius of a few tenths of a nanometer, they can reach every pore, defect and crevice on, or between, individual monofibrils. Nitrogen adsorption can thus help characterize the surface of asbestos fibers, although all particles in the sample (monofibers, dust and grit) contribute to the measured surface area.

V.2 - Dye adsorption in solution

The surface adsorption of colored organic molecules, i.e., organic dyes, may be used in a way similar to nitrogen gas adsorption. Typical organic dye molecules are approximately 10-100 fold larger than nitrogen molecules and, thus, will not readily penetrate in pores and crevices in the subnanometer size range. This may be informative with regards to the surface porosity of chrysotile fibers. Moreover, the adsorption of dye molecules can be measured directly in aqueous fiber slurries; this feature makes the method most interesting for on-line monitoring in wet-milling circuits.

V.3 - Infrared spectroscopy

Infrared spectroscopy consists in the quantitative measurement of the interaction of infrared light with matter. The frequency of the incident light is scanned and specific variations in absorption intensities by the samples are recorded. The frequencies of the absorption peaks are caracteristic of certain types of chemical bonds, and depend on the types of atoms and the type of motions occurring between them (stretching, bending or deformation). The data used in the present work have been obtained in the transmission mode, in which case the signal arises mainly from absorption in the bulk of the sample.

V.4 - Thermogravimetric analysis

A thermogravimetric analysis (TGA) essentially consist in the precise recording of the weight of a sample as its temperature is scanned from room temperature up to ∿1000°C. The weight losses due to the sequential decomposition of the various minerals can be detected and identified from the temperature at which the losses occur.

For example, magnesite decomposition to magnesium oxide ($MgCO_3 \rightarrow MgO + CO_2\uparrow$) releasing CO_2 gas at 450-600°C, can be discriminated from the chrysotile dehydroxylation process ($2Mg_3Si_2O_5(OH)_4 \rightarrow 3Mg_2SiO_4 + SiO_2 + 4H_2O\uparrow$) occuring over 600°C. The precise evaluation of the different mineral contents is a difficult task, but the presence or absence of some foreign materials commonly found in chrysotile can be easily established by this method. It must be noted, however, that magnetite is not detected by TGA analysis. The TGA data reported here have been recorded at a scanning rate of 20°C/min over a nitrogen stream flow of 50 ml/min.

V.5 - Optical microscopy and length-diameter sampling

The optical microscopy (OM) equipment listed in Table V.1 was used to produce high quality pictures of magnified asbestos samples. The fibers were first colored with an anionic blue dye (Acid Black was found to be an excellent dyeing agent) in order to enhance contrast. The colored fibers in a dilute aqueous slurry were then deposited on a Micropore filter; portions of the filter were selected and mounted permanently on glass slides. The filter material was rendered transparent by exposure to acetone vapor.

The slide mounted fibers were photographed under various magnifications to establish proper conditions for qualitative and quantitative studies. A 6"X10" high contrast print at 47X magnification was chosen for the measurement of fiber lengths and diameters; the measurements were carried out using a digitizing table. Each fiber measured was labelled numerically for later correlations between lengths and diameters. The data were first entered on an Apple IIe computer where they are given proper file format; they were then transferred to a IBM XT computer for later calculations and correlations.

In the fiber size measurement procedure, the following criteria were applied: all fibers touching two sides of the frames were eliminated; fibers touching the two other sides were assigned a length double to the length of the visible fragment. The length and diameter data were sorted by population classes and normalized distributions according to length and diameter were computed; the mean lengths and diameters and the standard deviations of the distributions were also evaluated. For each fiber sample, at least 400 fibers have been measured and satisfactory reproducibility was verified within different frames of the same fiber sample. Detailed methodologies, protocols and examples are presented in Appendix A: extensive data and calculations for the length and diameter distributions are listed in Appendix B.

V.6 - Scanning electron microscopy

The use of electron microscopy (i.e. image formed by reflected electrons rather than visible light) enables very high magnification under high resolution. Transmission electron microscopy (TEM) is mostly used to define contours and was initially envisaged to examine the shortest fiber (S) samples. It turned out, however, that the major fraction of S (wet-milled) fibers have length in excess of 50 μm, so TEM measurements were of little interest (they would be for detailed characterization of the fibers and dust particles in the size range 1-50 μm).

On the other hand, scanning electron microscopy (SEM) can provide clear pictures of the detailed morphology and opening of the chrysotile fiber bundles and was used extensively in the present work. SEM frames reported in later sections were mostly obtained under magnifications of 700X and 2200X.

V.7 - Kajaani Fiber Length Analyser

A Kajaani (FS-100) Fiber Length Analyzer was used to measure the length distributions of fibers in dilute aqueous suspensions. In this instrument, the suspension is aspirated from the bottom of a funnel-shaped, stirred container, and circulated through a thin (0.4 mm dia.) capillary. An optical device consisting of a light, condenser lenses and a series of photosensitive cells, enables the measurement of individual fiber lengths. The data are compiled by a microprocessor to yield fiber length populations and normalized distributions. Within a few minutes, the data for several thousand fibers can be accumulated providing excellent statistical results. The minimum detectable length is quoted as 50 μm (0.05 mm). A schematic description and detailed characteristics of the instrument is given in Appendix C.

VI - MATERIALS: Selection and physico-chemical analysis

We list in Table VI.1, the various chrysotile fiber products examined in this work. The study focussed mainly on different classes of dry-milled fibers commercially produced by Cassiar Mining Corp., and several wet-milled products also produced recently by Cassiar. Two other asbestos fibers from different locations were chosen for comparison purposes: a dry-milled 4T fiber from a Quebec producer and 10-mesh wet-milled fibers from Woodsreef, Australia.

TABLE VI.1: Sample designation, process type and origin of the asbestos fibers examined in this work

Grade	Milling process	Mine	Location
S	Wet	Cassiar Corp.	British Columbia
M	"	"	"
L	"	"	"
XL	"	"	"
FEED(1)	"	"	"
AZ	Dry	Cassiar Corp.	British Columbia
AY	"	"	"
AX	"	"	"
4T	Dry	N.A.	Québec
10-mesh	Wet	Woodsreef	Australia

(1) The "FEED" is the bulk wet-milled fiber product which is later classified into S, M, L and XL grades.

VI.1 - Elemental analysis

The results from an extensive composition analysis, carried out on selected samples, are presented in Table VI.2. Comparison between Cassiar AX and Quebec 4T grades, provides indications on variations in the chemical composition of chrysotile from different locations. The silica and magnesia contents, the main constituents of chrysotile, are quite similar; iron, which is usually the most abundant contaminant of chrysotile, is found at a significantly lower concentration in the Cassiar fibers than in the Quebec 4T fibers. If we further distinguish between the ferrous (Fe^{+2}) and ferric (Fe^{+3}) states for each grade, it appears that the former mainly accounts for differences in total iron levels. Significant differences also arises in some trace metals: comparing AX and 4T fibers, aluminium and nickel concentrations are found higher in AX fibers whereas calcium, potassium and carbon are lower.

TABLE VI.2: Elemental composition of wet-milled S and L fibers from Cassiar, dry-milled AX fibers from Cassiar and 4T fibers from Quebec (in weight %, unless specified otherwise)

Extraction	Wet-milled		Dry-milled	
Origin	Cassiar	Cassiar	Cassiar	Quebec
Grade	S	L	AX	4T
SiO_2	40.9	41.1	40.8	38.6
Al_2O_3	0.62	0.60	0.85	0.45
Fe_2O_{3tot} (1)	2.14	1.66	2.78	4.24
MgO	41.1	39.9	41.2	39.9
CaO	<0.02	<0.02	<0.02	0.13
Na_2O	<0.10	<0.01	<0.01	0.04
K_2O	<0.01	<0.01	<0.01	0.04
TiO_2	<0.01	<0.01	<0.01	0.01
MnO	0.05	0.04	0.06	0.08
P_2O_5	<0.01	<0.01	<0.01	<0.01
Volatiles (2)	14.9	15.0	14.7	15.3
FeO	0.24	0.20	0.40	0.90
Fe_2O_3	1.87	1.44	2.34	3.24
S	0.07	0.07	0.02	0.03
C_{tot} (CO_2)	0.53	0.35	<0.10	0.84
Cr	0.03	0.02	0.03	0.06
Co	43 ppm	37 ppm	51 ppm	38 ppm
Ni	0.14	0.14	0.16	0.058
V	18 ppm	40 ppm	29 ppm	5 ppm

(1) The total amount of Fe in the samples, is first expressed as Fe_2O_{3tot}. Distinction between ferrous and ferric oxidation states are made in the lower section (FeO and Fe_2O_3).

(2) Weight loss upon high temperature heating due mostly to dehydration and carbonate decomposition.

Comparison between wet-milled (S, L) fibers and AX fibers reveal that the former exhibit a lower total iron level and that the reduction bears equally on the levels of ferrous and ferric iron. This most probably results from the removal of magnetite particles in the wet-milling process.

VI.2 - Thermogravimetric analysis curves

Typical TGA curves obtained for the wet-milled FEED and S fibers, and the dry-milled AX fibers are illustrated in Fig. VI.1a-c. These curves essentially yield the weight loss as recorded over the temperature scanning range. Up to 300°C, there is no significant decomposition and any weight loss observed in the low temperature region is due to adsorbed molecules, mainly water. The first dehydroxylation process occurs in the 270°C to 460°C range and is associated with brucite dehydration ($Mg(OH)_2$). No significant levels of brucite were detected in any of the Cassiar fibers. In the range 450°C to 600°C, the CO_2 loss by magnesite ($MgCO_3$) is usually apparent. The major weight loss occuring in the 600°C to 750°C region is due to chrysotile dehydroxylation into forsterite (Mg_2SiO_4) **(the detailed shape of this peak depends, in part, on the physical state of the sample (granulometry, compaction) and should therefore not be interpreted unless a detailed assessment of the influence of the physical parameters has been carried out)**. Finally, the presence of calcite ($CaCO_3$) can be detected from its CO_2 loss up to 900°C. Since the elemental analysis showed very low calcium levels in the Cassiar fibers and substantial carbon levels in S and L grades, the 0.64% weight loss observed with the S fibers between 300°C and 500°C is likely due to magnesite. The TGA data obtained for other Cassiar grades are presented in Appendix D.

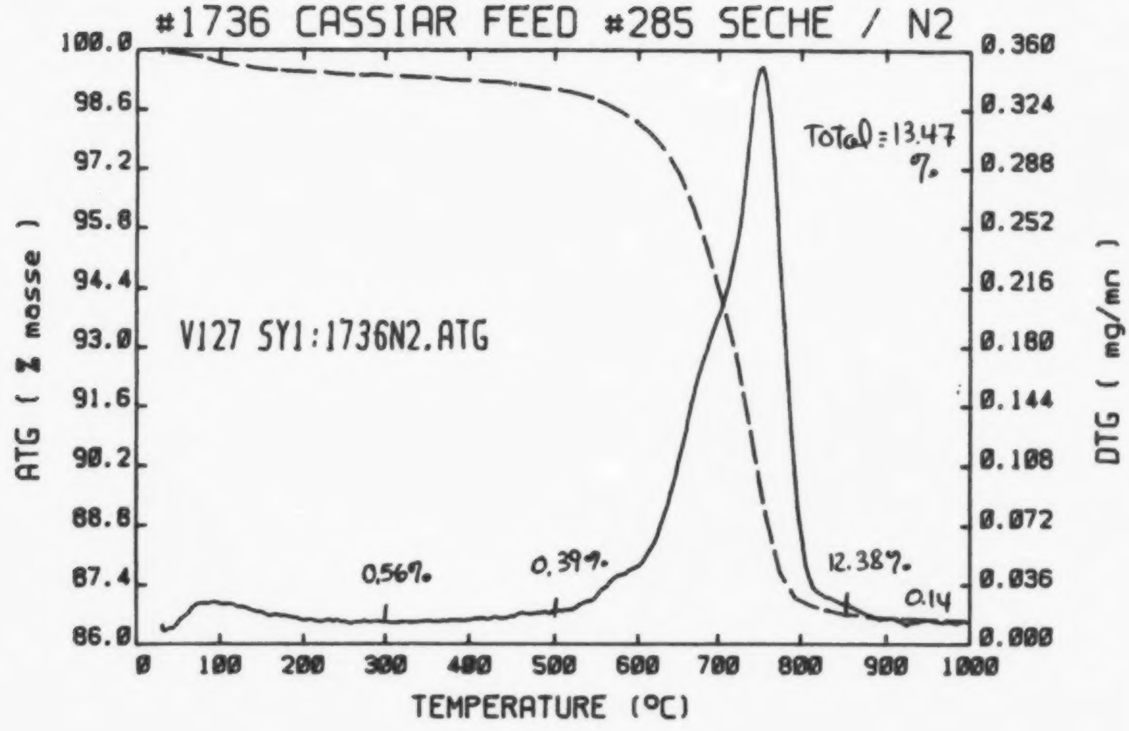


FIG. VI.1a) TGA for wet-milled FEED fibers. Thermogravimetric Analysis curve of wet-milled FEED fibers; dotted line: % weight change as function of temperature; full line: rate of weight loss in mg/min. The weight loss data entered along the curve refer to the temperature interval between two consecutive markers.

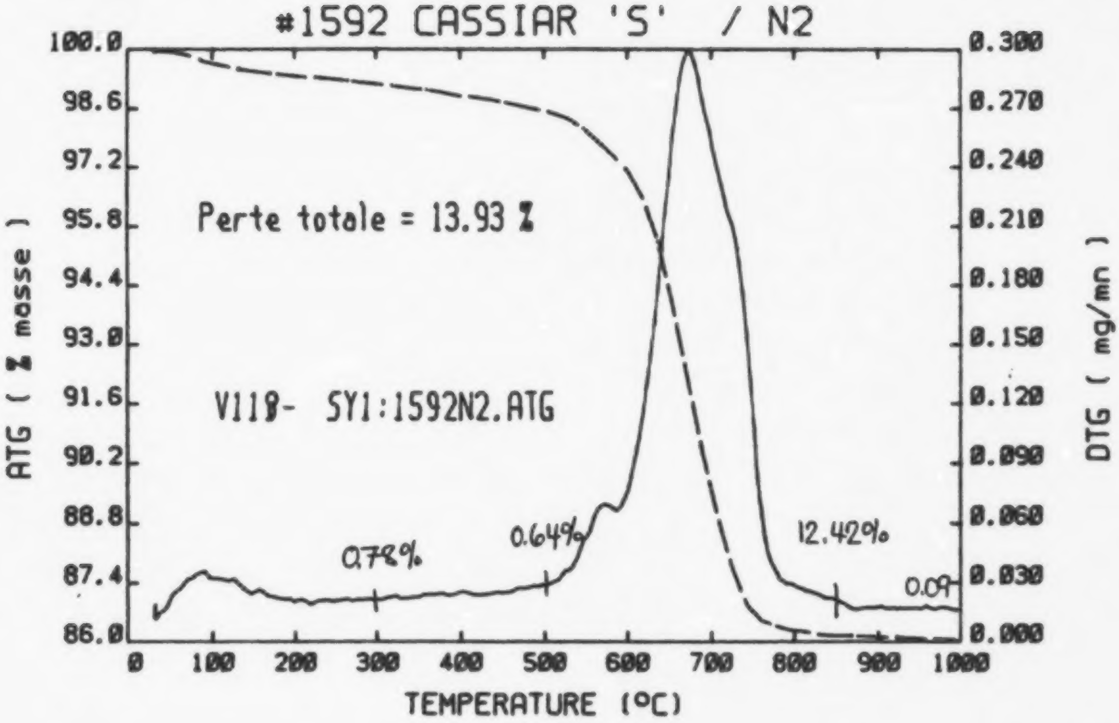


FIG. VI.1b) TGA curve for wet-milled S grade. (For details see FIG. VI.1a).

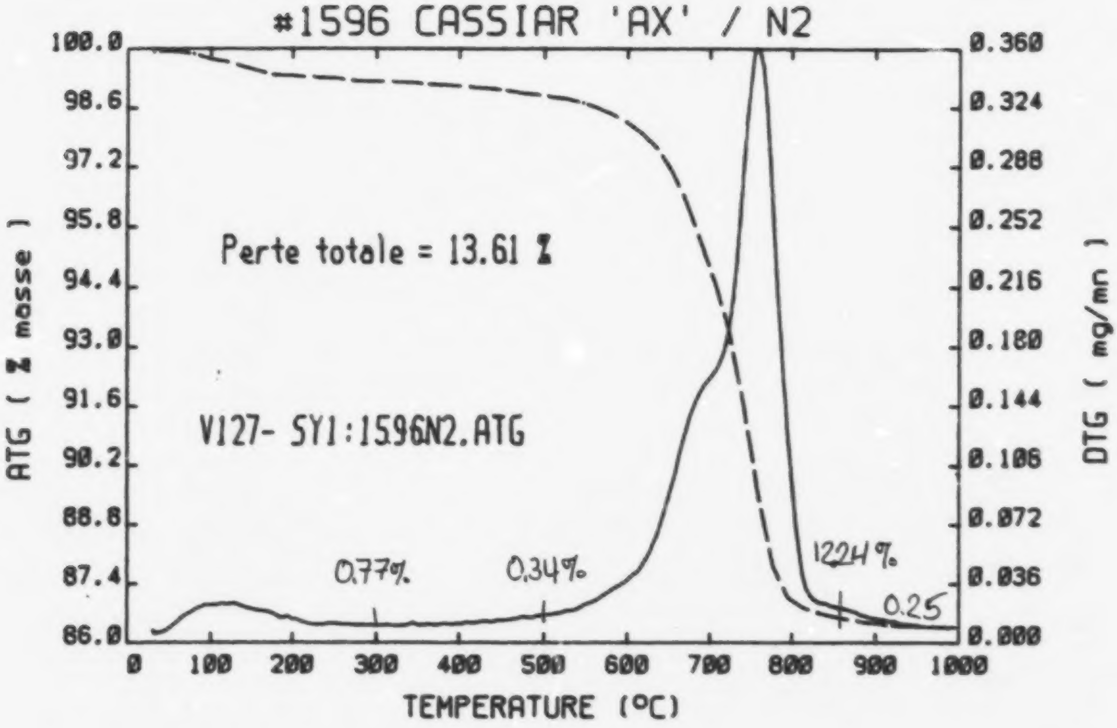


FIG. VI.1c) TGA curve for dry-milled AX grade.
(For details see FIG. VI.1a).

VI.3 - Infrared spectra

The infrared (IR) spectra of the wet-milled FEED and S fibers, and of the dry-milled AX fibers are presented in Fig. VI.2a-c; these spectra are typical of the main constituents of chrysotile asbestos. The chrysotile IR spectrum may be separated in four main regions, labelled here A to D. The higher frequency region (A) shows two fine peaks at 3690 cm^{-1} and 3646 cm^{-1} which originate from the stretching vibrations of O-H groups in the brucite layer. The (B) region corresponds to a large range of frequencies below 3600 cm^{-1} where physically adsorbed molecules can be observed; for example, surface adsorbed water contributes a weak and broad adsorption peak around 3300 cm^{-1}. In the following (C) region, a strong triplet at 1080, 1021 and 956 cm^{-1} and two other peaks at 658 and 603 cm^{-1}, can be observed which are characteristic of the different vibrational modes of Si-O bond encountered in the tetrahedral Si_2O_5 layer. The remaining low frequency (D) region reflects deformation lattice vibrations, the exact assignation of which remains uncertain.

Examination of the various spectral regions shows that the FEED, S and AX fibers exhibit very similar IR spectra typical of all chrysotile materials. Minor differences between the spectra of AX and FEED fibers suggest a higher level of surface contaminants for the AX fibers (region B at $\sim$ 2200 cm^{-1}) and a higher content of adsorbed water with the FEED fibers. The IR spectra of other Cassiar fibers are given in Appendix E.

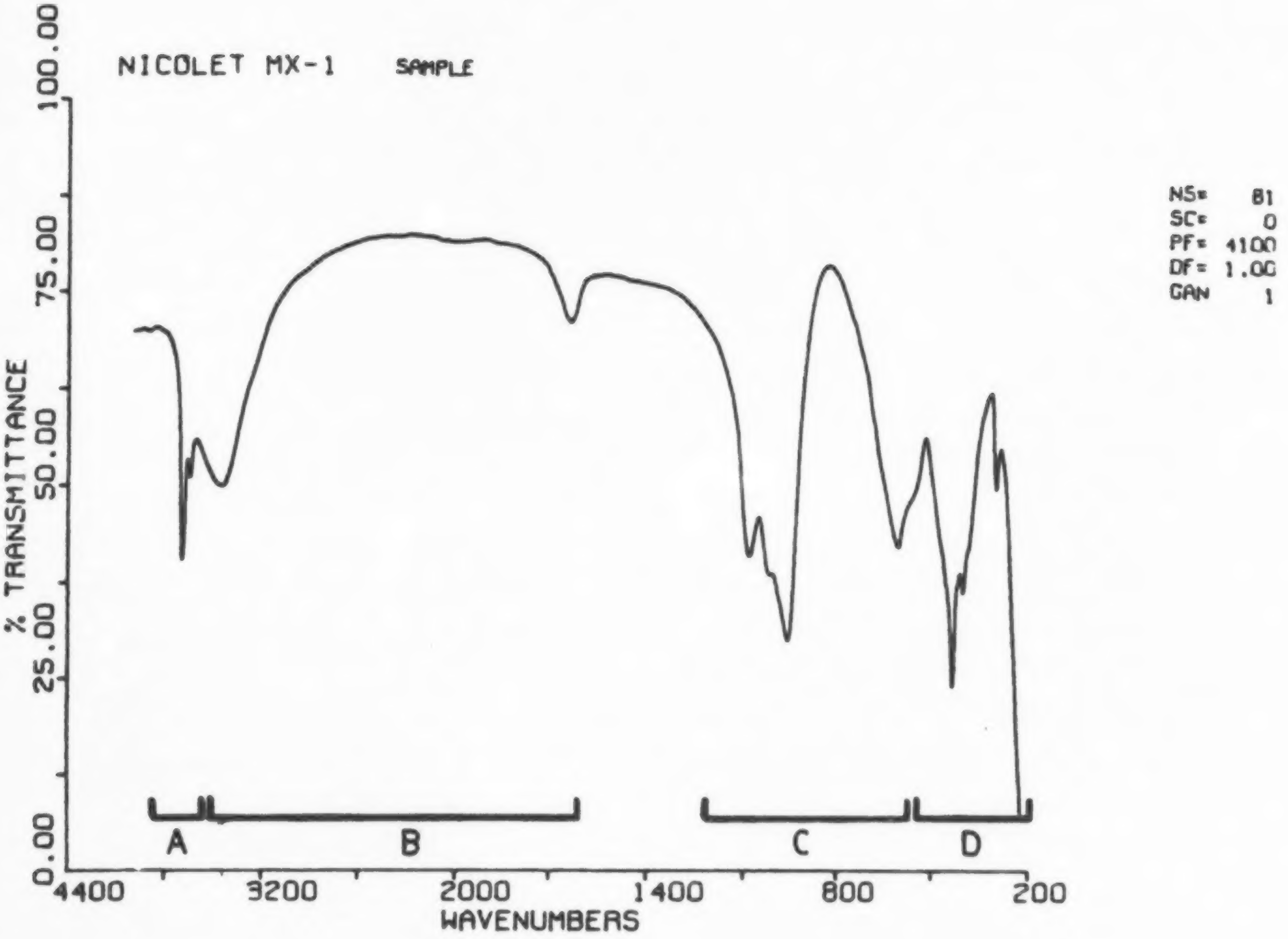


FIG. VI.2a) IR spectrum of wet-milled FEED fibers.

A: 3600-3700 cm^{-1}: 3690 and 3646 cm^{-1}: hydroxide groups of brucite layers
B: 1600-3600 cm^{-1}: 3300 and 1650 cm^{-1}: adsorbed water; organic contaminants
C: 600-1100 cm^{-1}: 1018, 1021 and 956 cm^{-1}: characteristic vibrational triplet of SiO_2 tetrahedra in chrysotile; 658 and 603 cm^{-1}: SiO_2 in chrysotile
D: below 600 cm^{-1}: low frequency modes, assignment uncertain

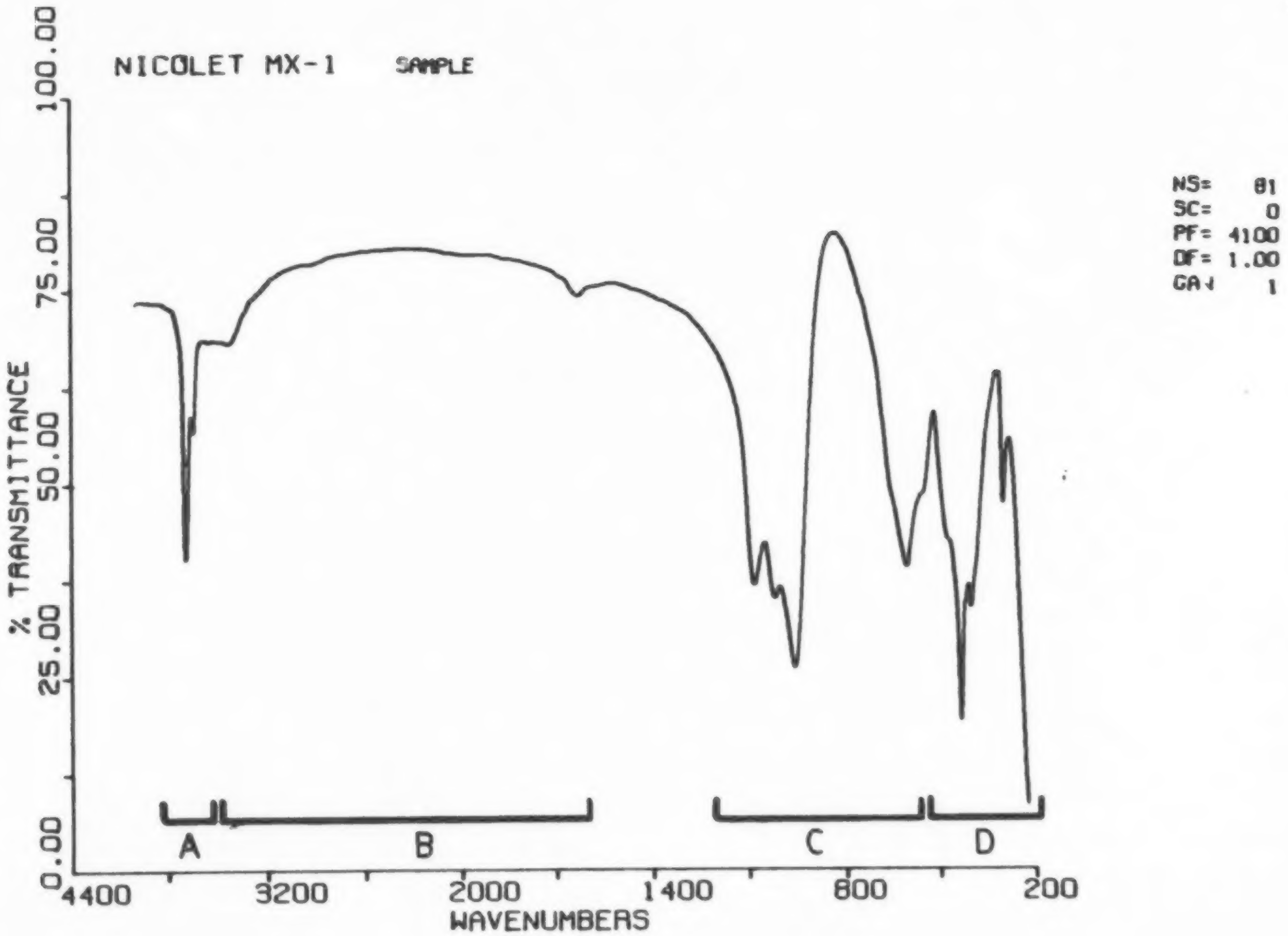


FIG. VI.2b) IR spectrum of wet-milled S grade.

A: 3600-3700 cm^{-1} (for significance of
B: 1600-3600 cm^{-1} absorption peaks,
C: 600-1100 cm^{-1} see Fig. VI.2a)
D: below 600 cm^{-1}

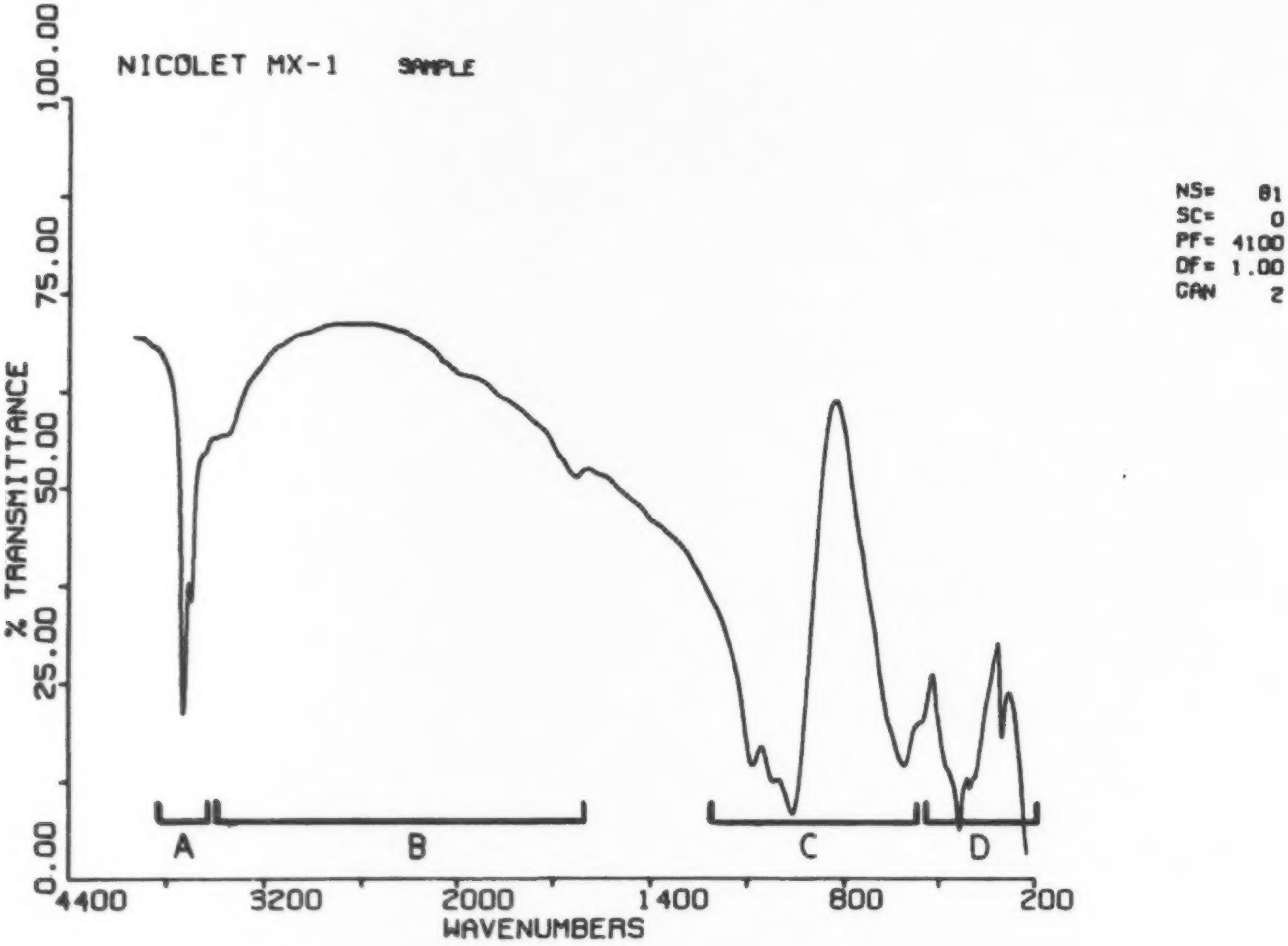


FIG. VI.2c) IR spectrum of dry-milled AX grades.

A: 3600-3700 cm^{-1}
B: 1600-3600 cm^{-1}
C: 600-1100 cm^{-1}
D: below 600 cm^{-1}

(For significance of absorption peaks, see Fig. VI.2a)

VI.4 - Concluding remark

The results derived from the various physico-chemical analysis presented above first confirm that all the Cassiar fiber products studied here (FEED, S, M, L, XL, AX, AY and AZ) are indeed chrysotile fibers with relatively low levels of the contaminants commonly found in chrysotile asbestos.

The iron content of the dry-milled fibers which is somewhat lower than that found in other Canadian chrysotile fibers, appears further reduced by the wet-milling process. Since both iron oxides (FeO and Fe_2O_3) are found to decrease simultaneously, we must conclude that the lower iron content in the wet-milled fibers is due to elimination of magnetite (Fe_3O_4) particles in the wet fiber circuit.

VII - IMPORTANT QUALITATIVE OBSERVATIONS FROM MICROSCOPY

In addition to providing the basic fiber dimensional parameters (ℓ, d, ℓ/d) and distributions of these parameters, the optical and electron microscopy techniques yield a variety of important qualitative results. The most important features noted throughout this work are reported below, first for the bulk commercial materials (**bulk**) and second for the opened fibers (**Pallmann**).

VII.1 - Qualitative features of bulk fibers

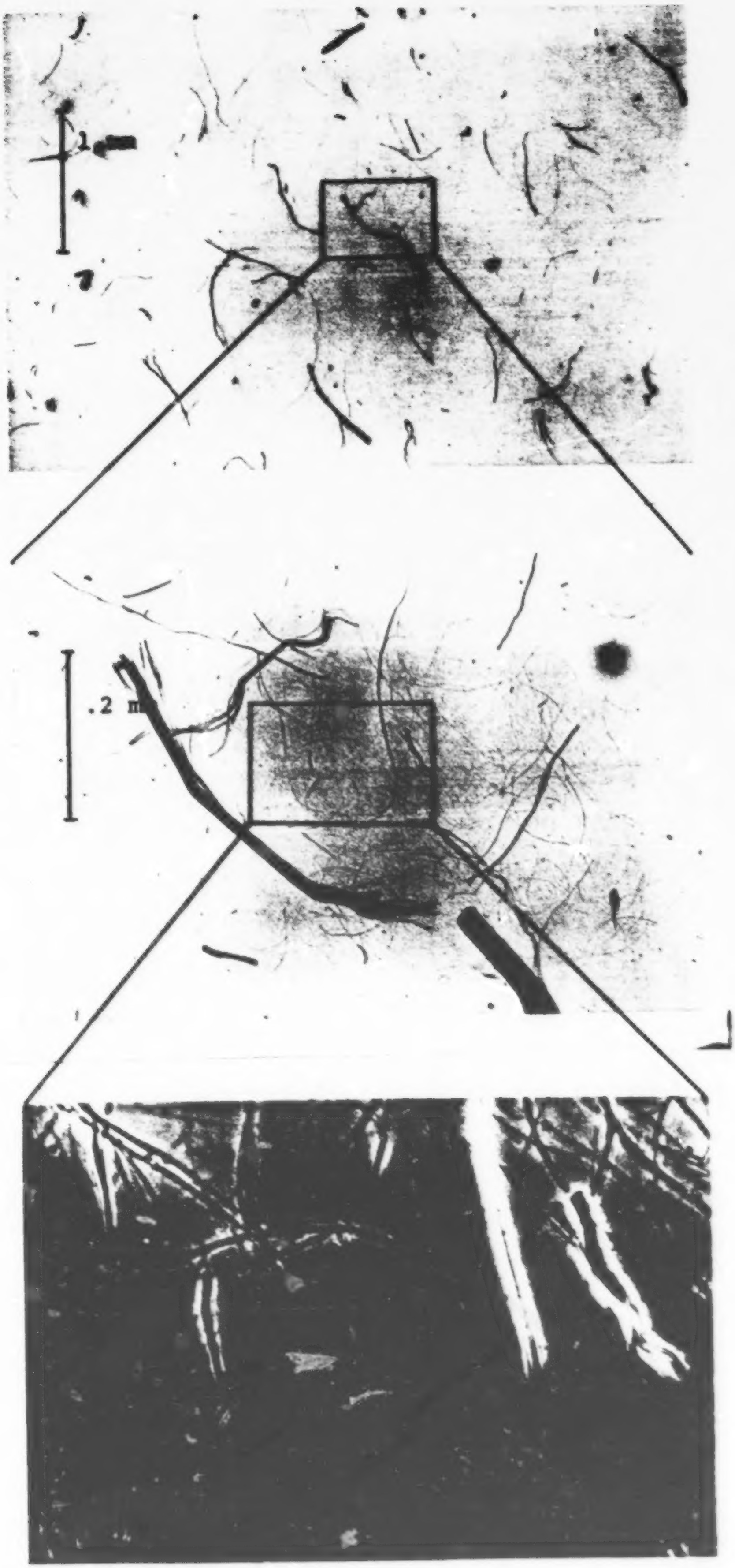
VII.1.a - Preliminary observations from optical microscopy

We reproduce in Fig. VII.1a-c, OM photographs showing representative sections of slide mounts under 22X, 140X and 540X magnification of the wet-milled S fibers and of the dry-milled AY and Quebec 4T fibers. The field retained upon increasing magnification is shown as an insert in the lower magnification photographs.

Comparing the frames of S fibers (a) with those of AY (b) and Quebec 4T (c) fibers, it is immediately apparent that the wet-milled S fibers contain much fewer non-fibrous particles (grit, dust); at the onset, the wet-milled fibers are thus seen to be cleaner than dry-milled fibers of the same origin (wet-milled S vs dry-milled AY).

Further comparison among Fig. a and b also shows that the wet-milled S fibers are more homogeneous (less bundles, spicules) than dry-milled fibers.

Detailed examination of photographs taken under progressive magnification reveals, at 540X, a large number of very fine fibers, especially with the S fibers (a). These thin fibers, (with diameters in the range 0 to 3 μm) are not included in our fiber size distribution analysis

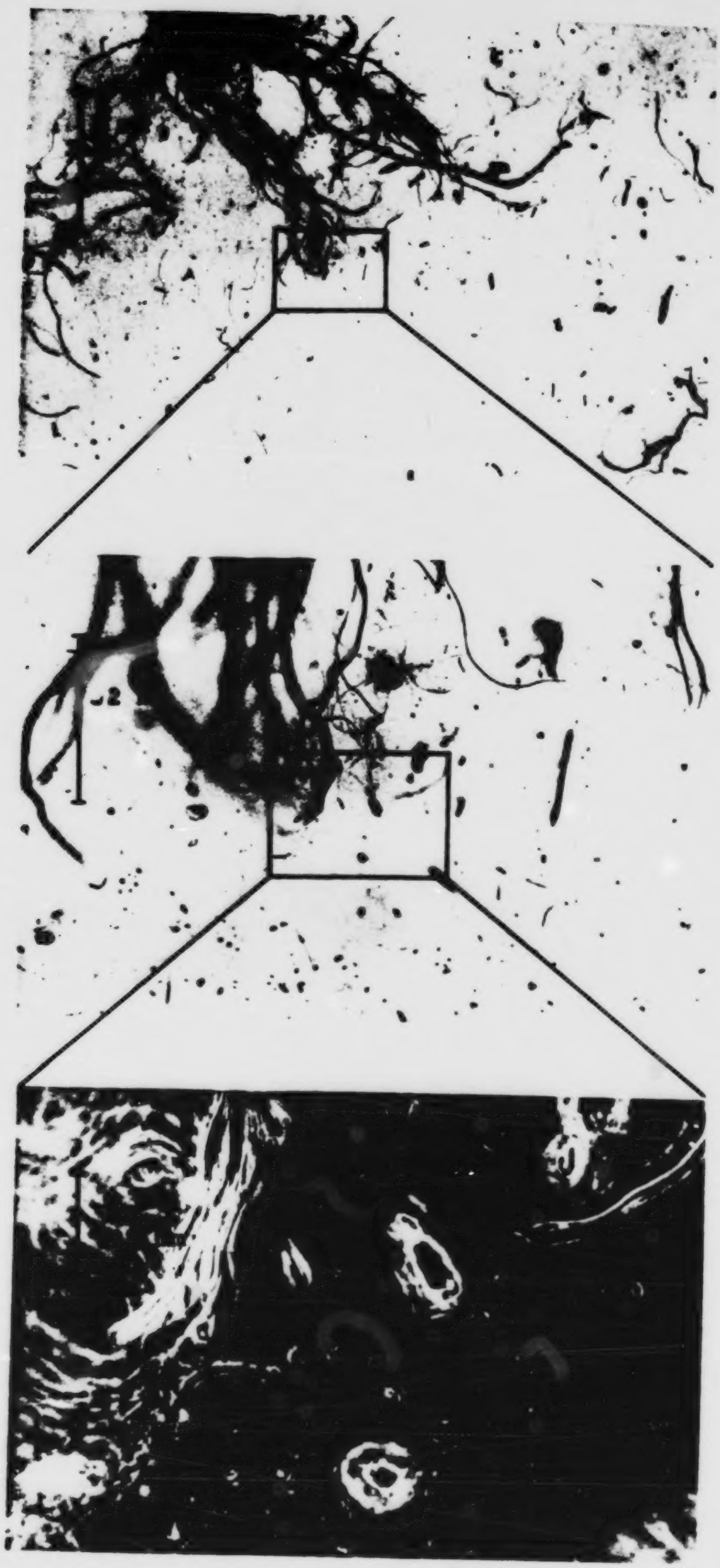


22X
(Bar = 1 mm)

140X
(Bar = .2 mm)

540X
(Bar = .05 mm)

FIG. VII.1a) Optical microscopy frames of wet-milled S fibers at different magnifications



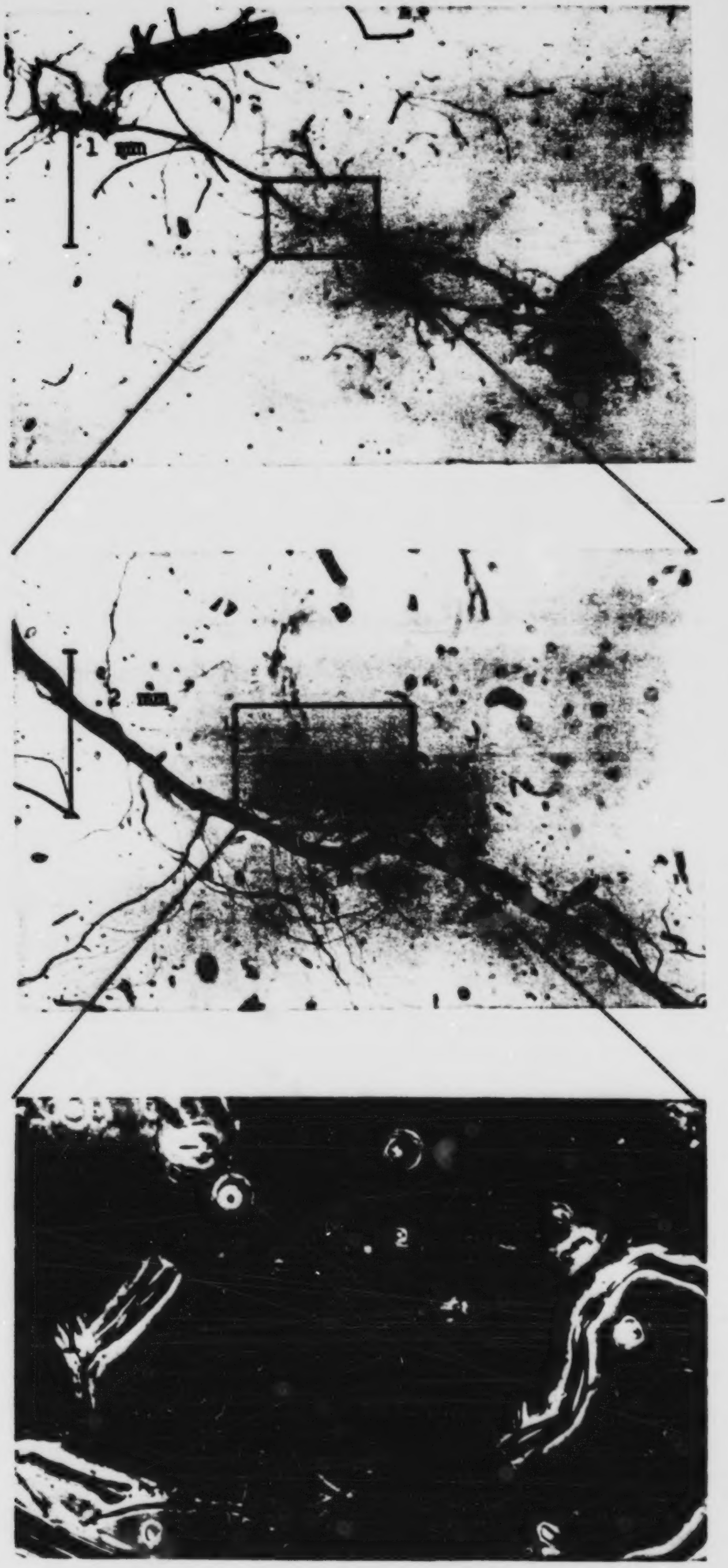
22X
(Bar = 1 mm)

140X
(Bar = .2 mm)

540X
(Bar = .05 mm)

FIG. VII.1c) Optical microscopy frames of Quebec 4T fibers
at different magnifications

FIG. VII.1b) Optical microscopy frames of dry-milled AY fibers at different magnifications



22X
(Bar = 1mm)

140X
(Bar = .2 mm)

540X
(Bar = .05 mm)

which was carried out at 47X. Such fine fibers are not expected to contribute significantly to the total mass of a given sample, nor to reinforcement of cement in A/C products; nonetheless, they may strongly influence some of the fiber tests, for example surface area and drainage.

VII.1.b - Scanning electron microscopy observations

In Fig. VII.2a-d, we illustrate scanning electron microscopy (SEM) photographs obtained at 700X and 2200X for the following fibers: a: bulk wet-milled (FEED) fibers; b: short wet-milled fibers (S); c: long wet-milled fibers (L); d: dry-milled AY fibers.

A comparison of the low magnification (700X) photographs for the S and AY fibers confirms the greater cleanness (low dust and grit contents) and greater homogeneity of the S fibers compared to the AY fibers. (The 700X SEM magnification is comparable to the highest OM 540X magnification on Fig. VII.1). A detailed examination of the SEM photographs leads to the following observations:

- the bulk wet-milled fibers (FEED) appear more heterogeneous (fiber size, shape and texture) than the S or L fibers;

- as noted from optical microscopy examination, the S fibers exhibit the greatest homogeneity in fiber sizes, shapes and textures; L fibers appear intermediate between the S and FEED fibers;

- the fiber "bundles" which are seen in the wet-milled products (FEED, S, L) exhibit a "loosely bonded" texture, in contrast to the "tight packing" appearance of bundles in the dry-milled AY fibers.

FIG. VII.2 (following pages)

Scanning electron microscopy (SEM) frames obtained for several wet- and dry-milled chrysotile fibers from Cassiar.

Upper frame : magnification: ∿700X
vertical bar : 100 μm
Lower frame : magnification: 2100X
vertical bar : 10 μm

a) Wet-milled fiber product
b) Wet-milled S fibers
c) Wet-milled L fibers
d) Dry-milled AY fibers

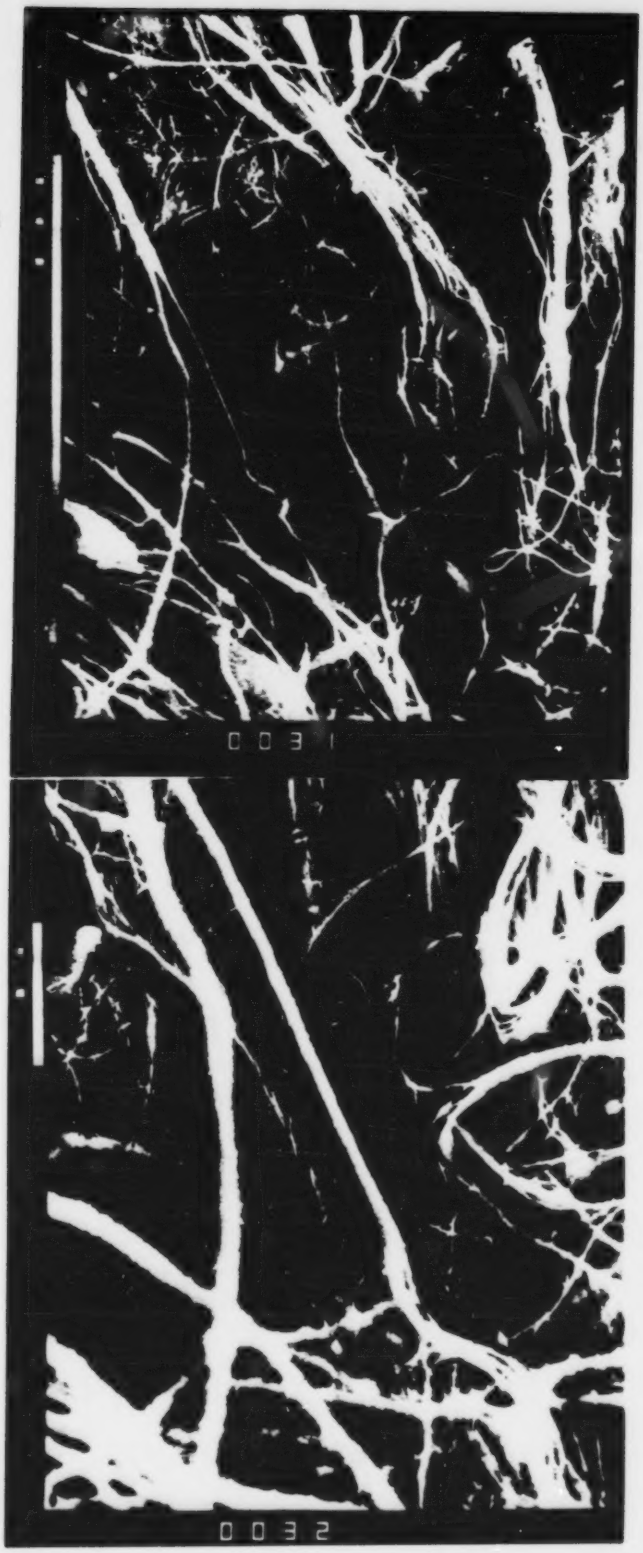

100 µm
720X
0031
10 µm
2100X
0032

FIG. VII.2a) SEM frames obtained with bulk wet-milled chrysotile fibers (FEED) from Cassiar

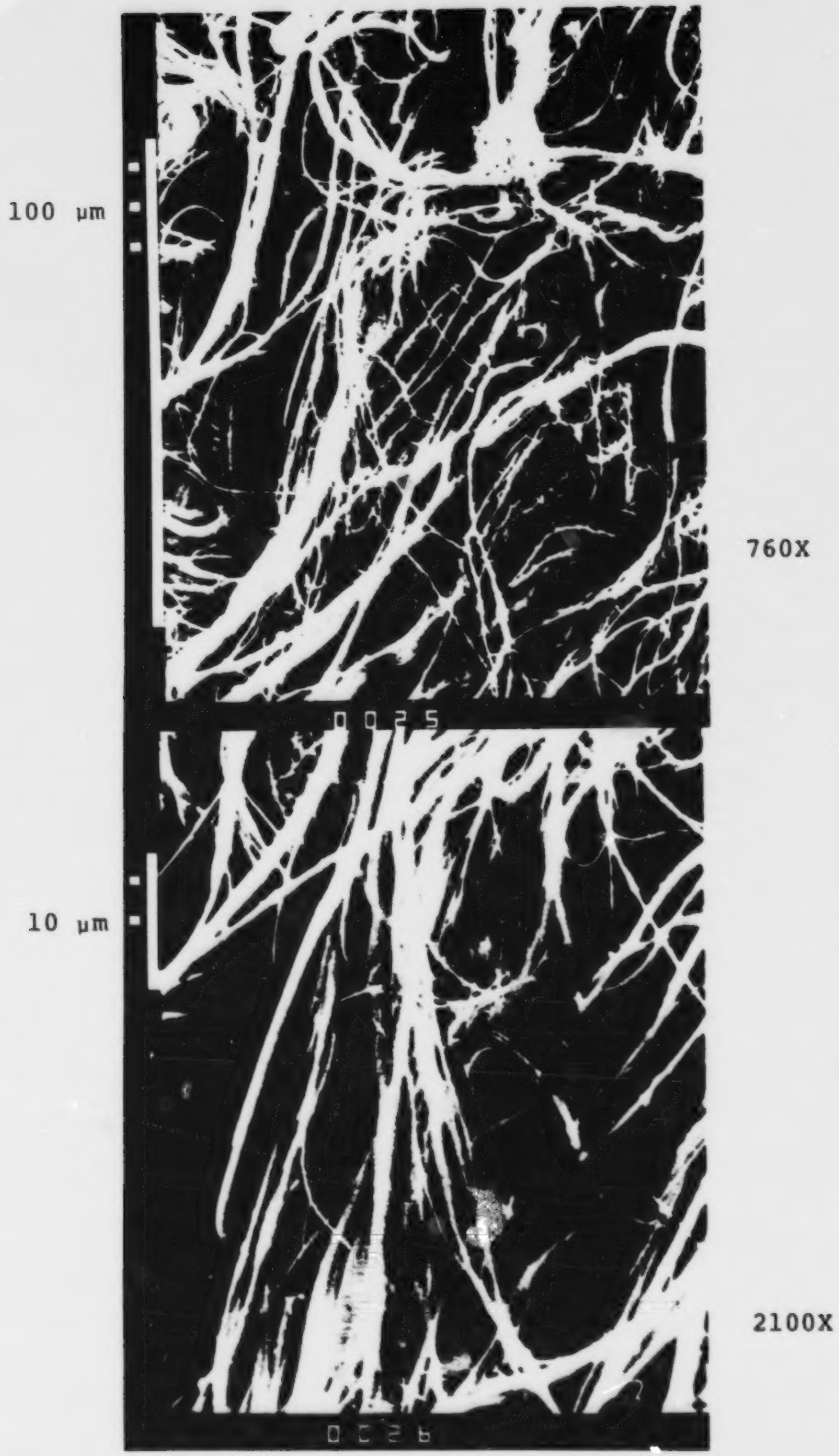

100 µm
760X
0025
10 µm
2100X
0026

FIG. VII.2b) SEM frames obtained with wet-milled classified short fibers (S) from Cassiar

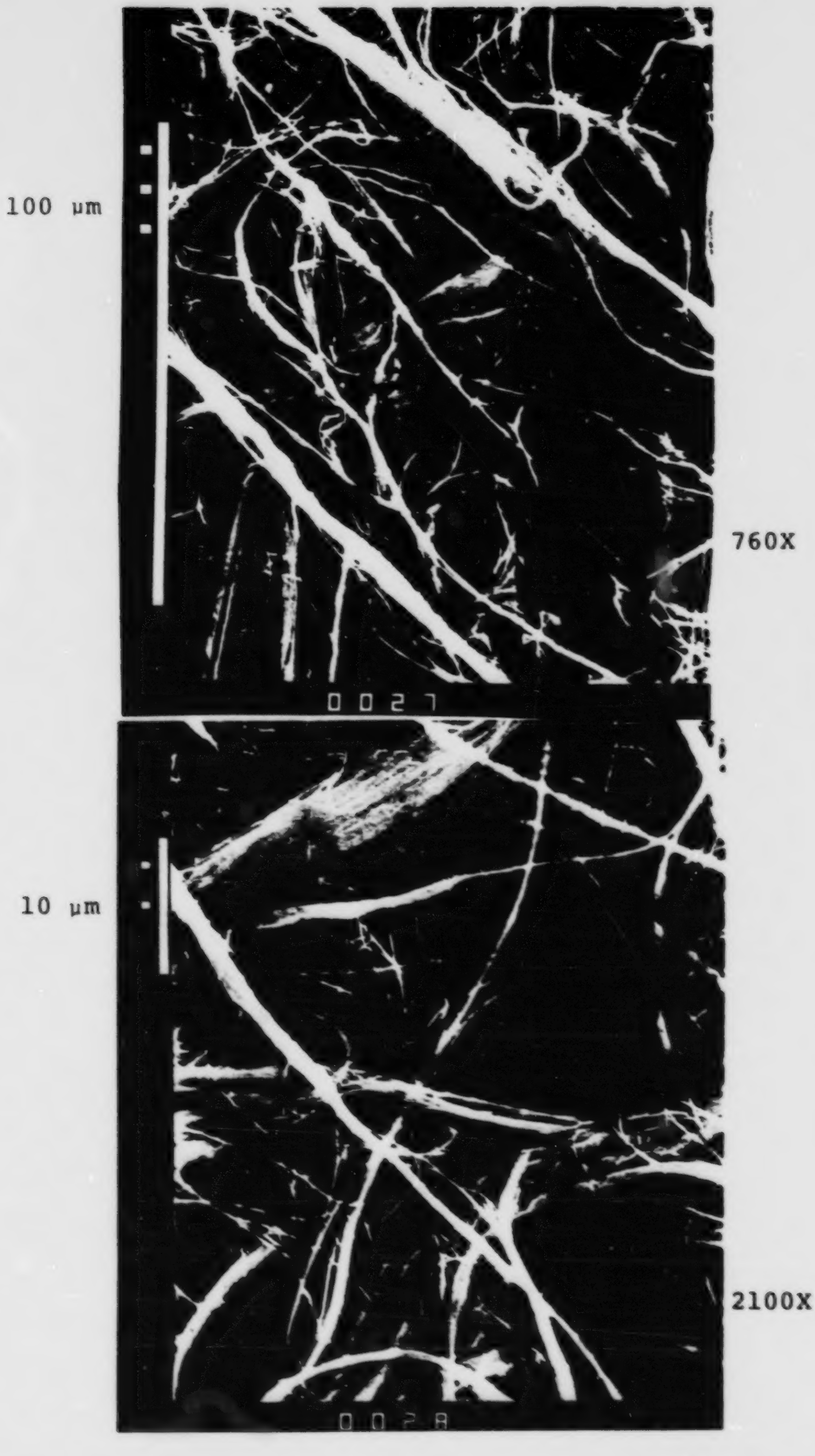

100 µm
760X
0027
10 µm
2100X
0028

FIG. VII.2c) SEM frames obtained with wet-milled classified long fibers (L) from Cassiar

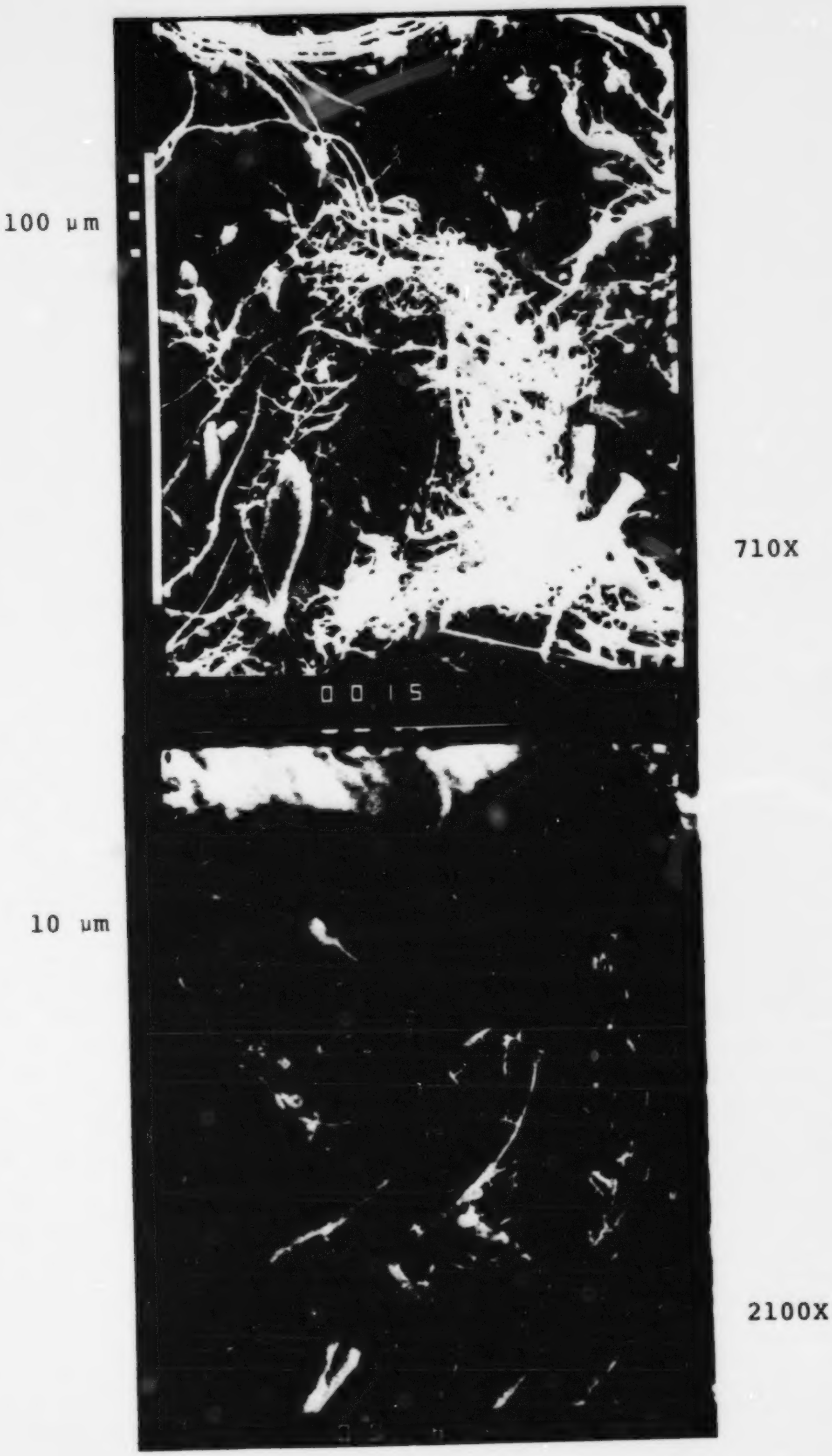

100 µm
710X
00 15
10 µm
2100X

FIG. VII.2d) SEM frames obtained with dry-milled AY fibers from Cassiar

VII.1.c - Optical microscopy frames for the various fiber types

We illustrate in Fig. VII.3a-k optical microscopy photographs taken at 47X magnification of the various fiber products examined here. The fiber length and diameter distribution reported in later sections were obtained from sets of these photographs.

The observations noted above from OM and SEM microscopy studies may be further visualized in Fig. VII.3a-k. For example, examination of the series of frames for S, M, L and XL fibers (a-d) shows an increasing amount of thick and compact bundles in the order S to XL. The apparent average length increases in the same order, while the occurence frequency of thin fibers decreases; heterogeneity of sizes, shapes and textures also appears to increase in the order S to XL. The bulk wet-milled FEED fibers (e, f), from which the S, M, L and XL grades were obtained, appears, again, as the most heterogeneous.

The dry-milled AZ, AY and AX Cassiar fiber samples illustrated in Fig. VII.3 (h, i, j) clearly exhibit higher dust-grit contents than the wet-milled fiber products. In contrast to the latter, thick and compact bundles are frequently apparent in the dry-milled samples; such bundles have odd shapes (bends, kinks, splittings) which makes it difficult to give a representative illustration of these fiber products. Hence, distinction between the AZ, AY and AX fibers cannot be reliably established on the basis of a single frame (the occurence of these bundles also complicate the statistical analysis of length and diameter distribution).

FIG. VII.3 (following pages)

Optical microscopy (OM) pictures obtained from wet-milled Cassiar and Woodsreef, and dry-milled Cassiar and Quebec 4T bulk chrysotile fibers. The approximate surface loading of the filter used is given in $\mu g/cm^2$.

Magnification: 47X; bar: 1 mm.

a) S (wet-milled)
b) M " "
c) L " "
d) XL " "
e) FEED " "
f) FEED " "
g) Woodsreef -10-mesh (wet-milled)
h) AZ (dry-milled)
i) AY " "
j) AX " "
k) Quebec 4T " "

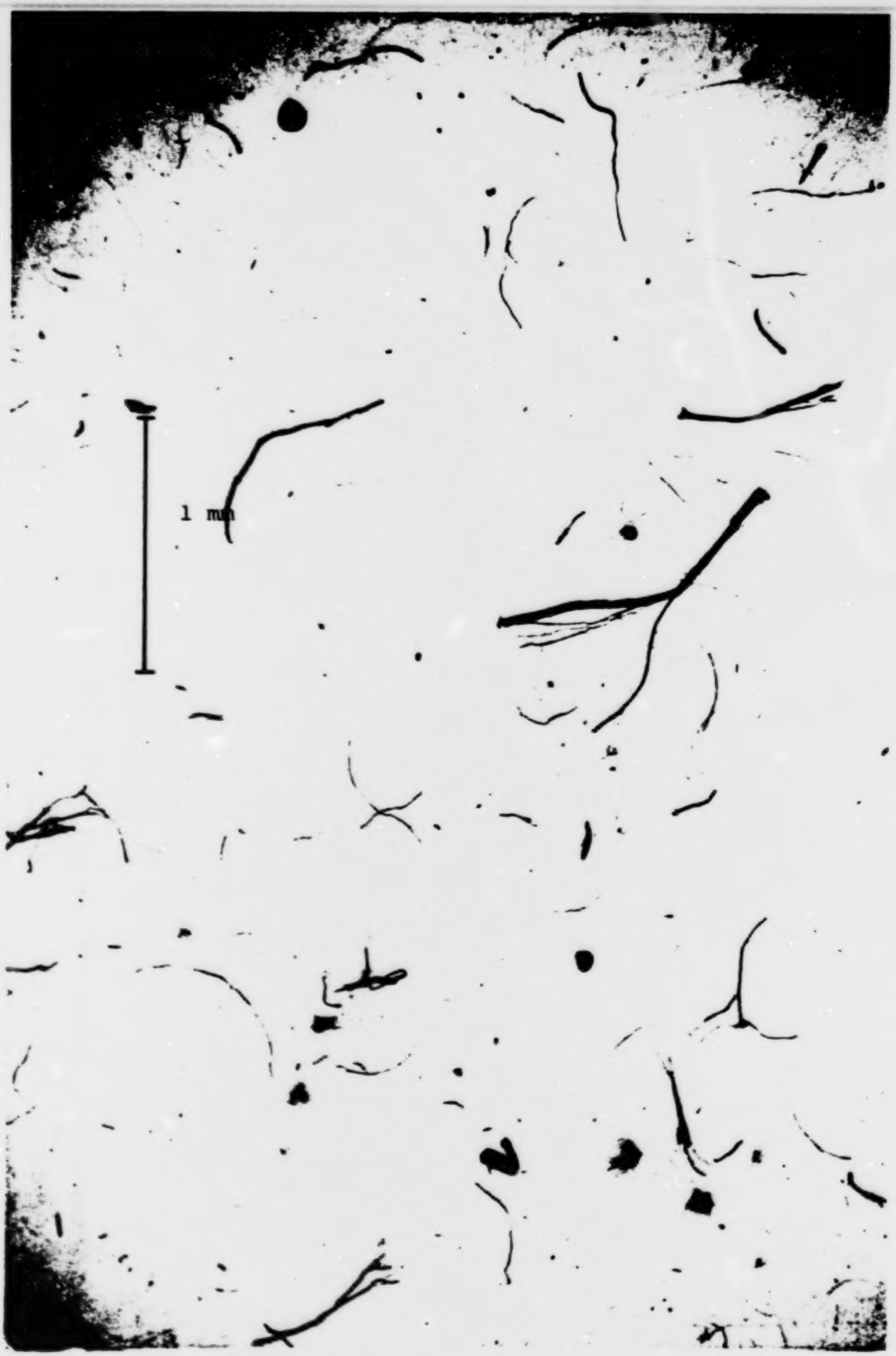


FIG. VII.3a) Cassiar wet-milled S fibers: filter loading: 25 μg/cm^2

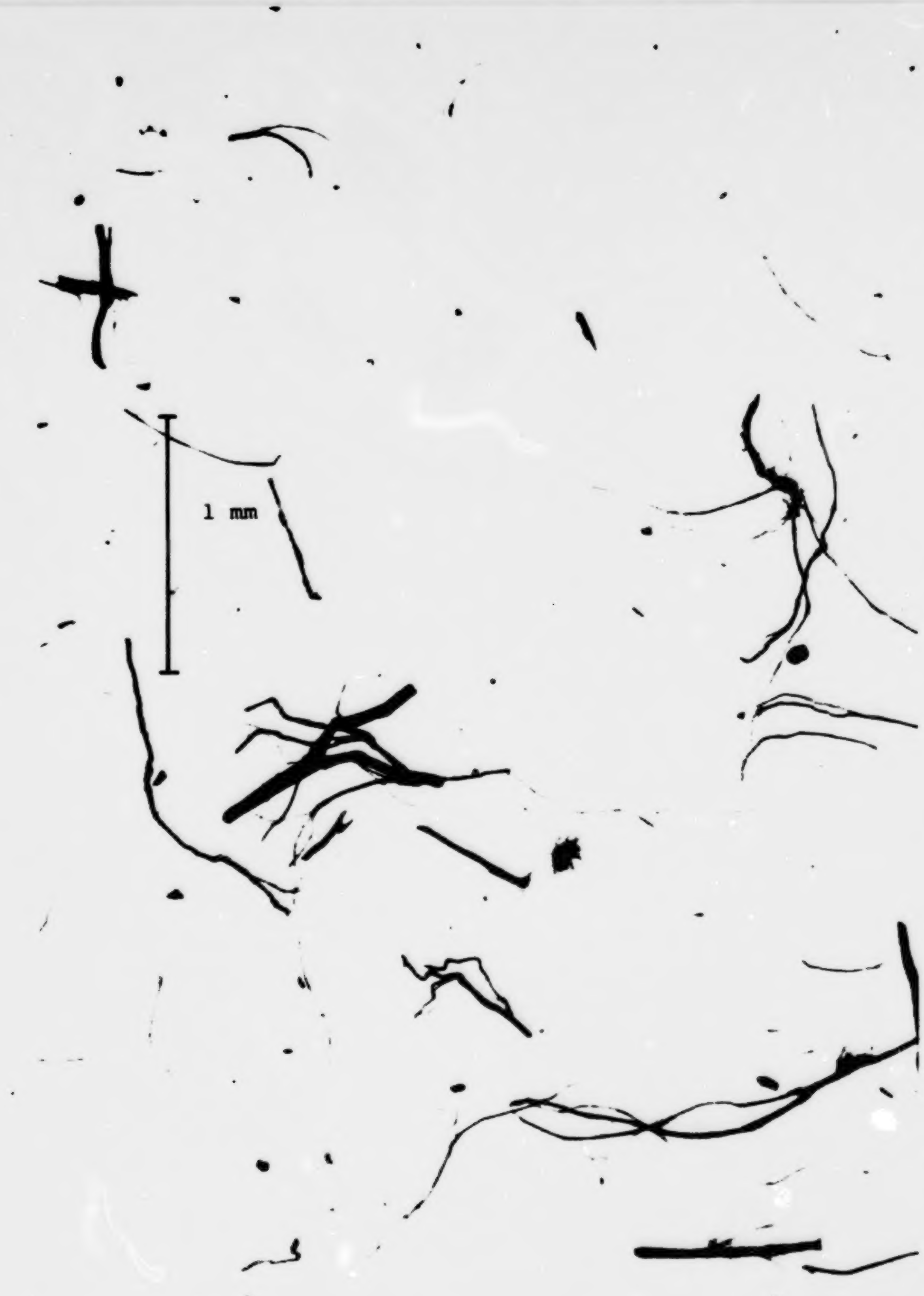


FIG. VII.3b) Cassiar wet-milled M fibers; filter loading: 50 μg/cm^2

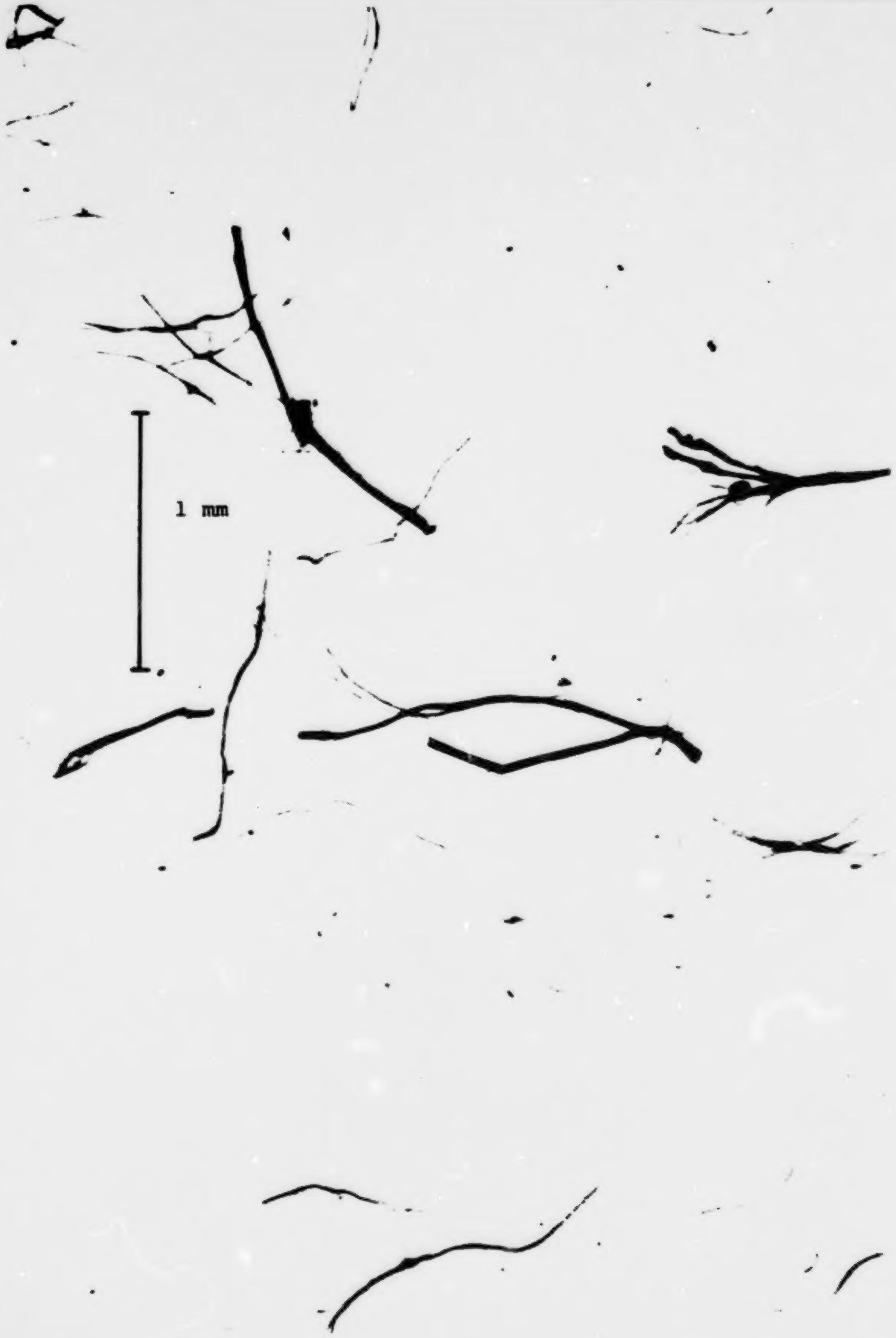


FIG. VII.3c) Cassiar wet-milled L fibers; filter loading: 50 µg/cm^2

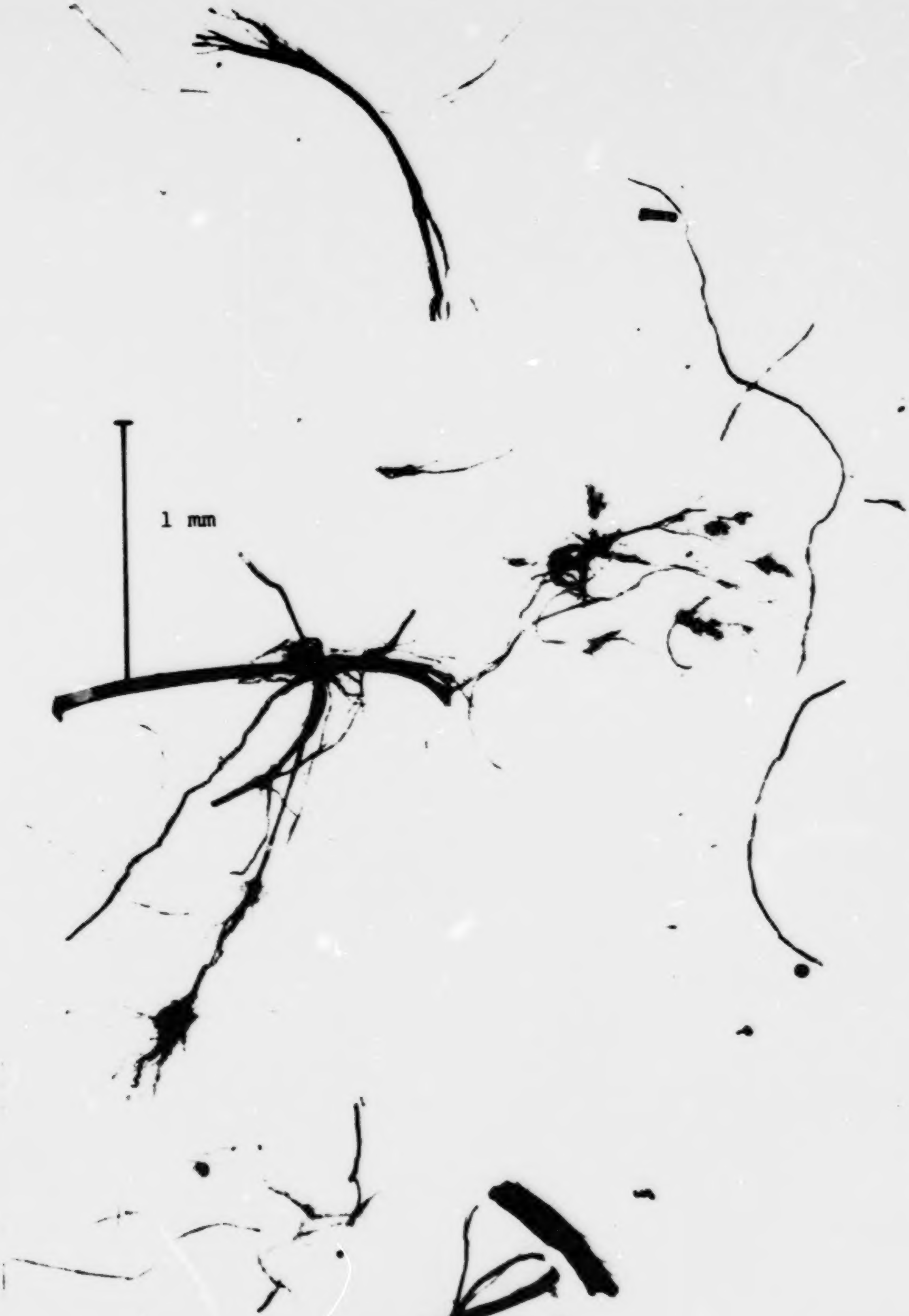


FIG. VII.3d) Cassiar wet-milled XL fibers; filter loading: 50 μg/cm^2

FIG. VII.3e) Cassiar wet-milled FEED fibers; filter loading: 25 μg/cm^2

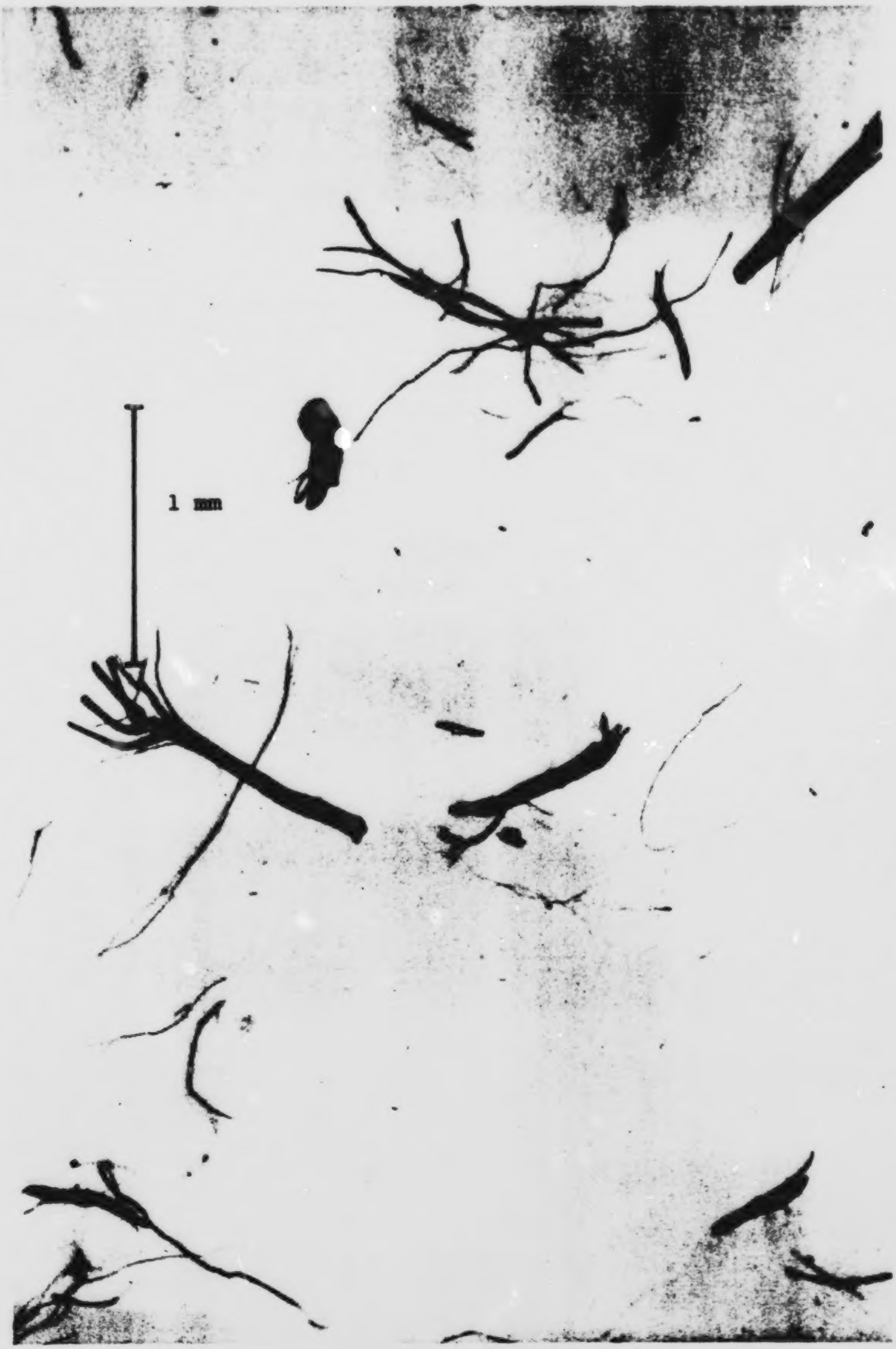


FIG. VII.3f) Same as Fig. VII.3e), different field

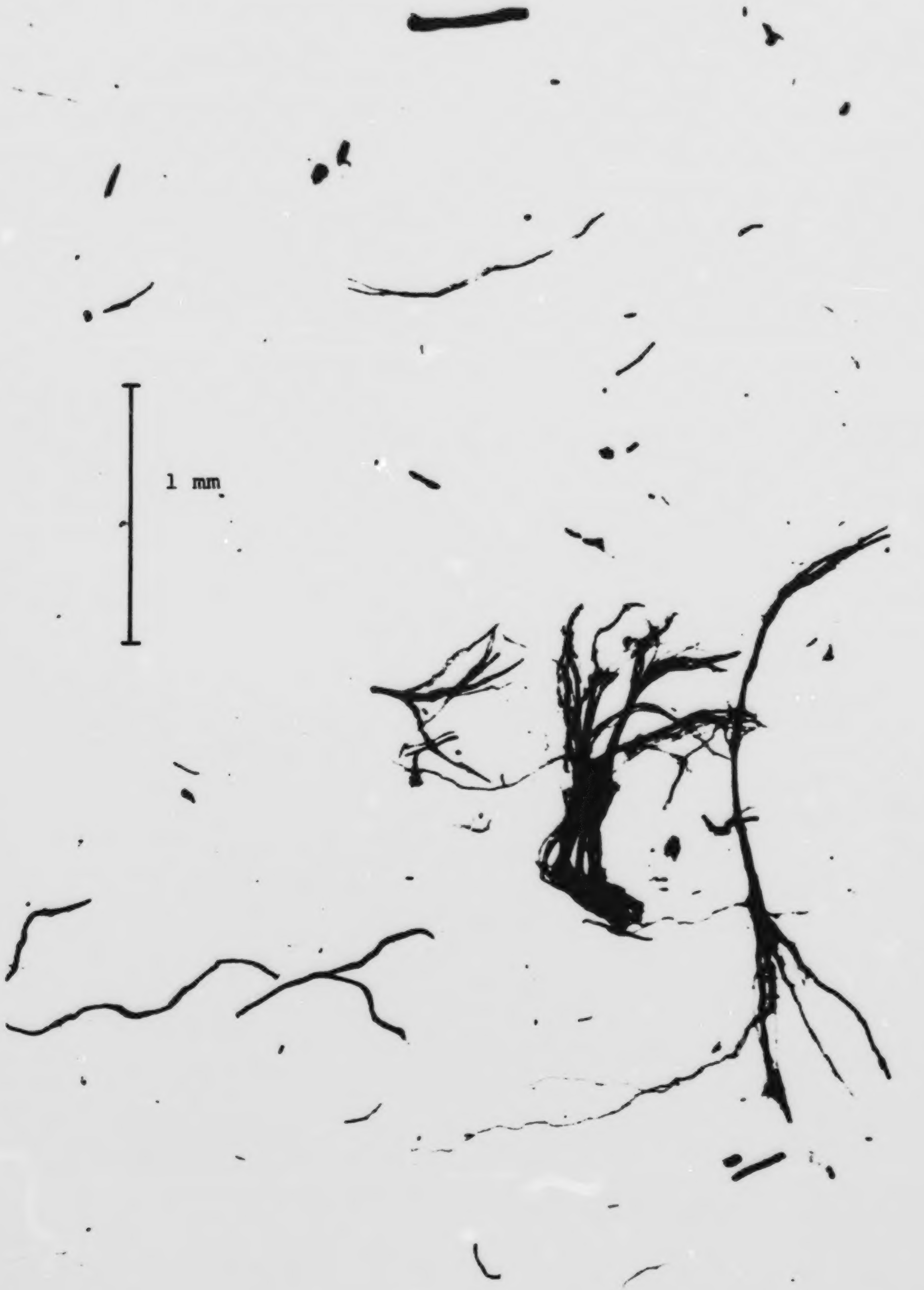


FIG. VII.3g) Woodsreef 10-mesh wet-milled fibers; filter loading: 50 μg/cm^2

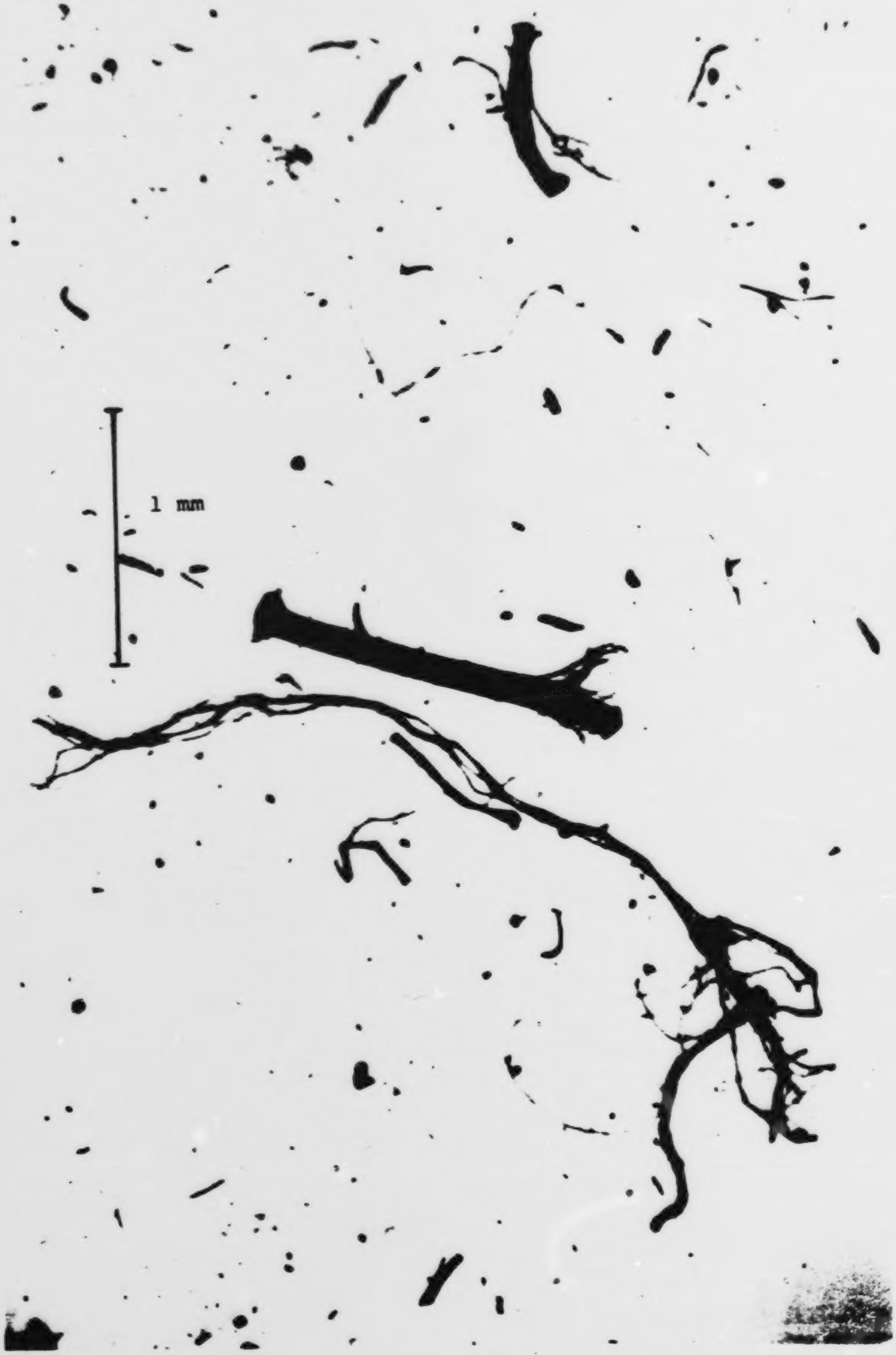


FIG. VII.3h) Cassiar dry-milled AZ fibers; filter loading: 100 μg/cm^2

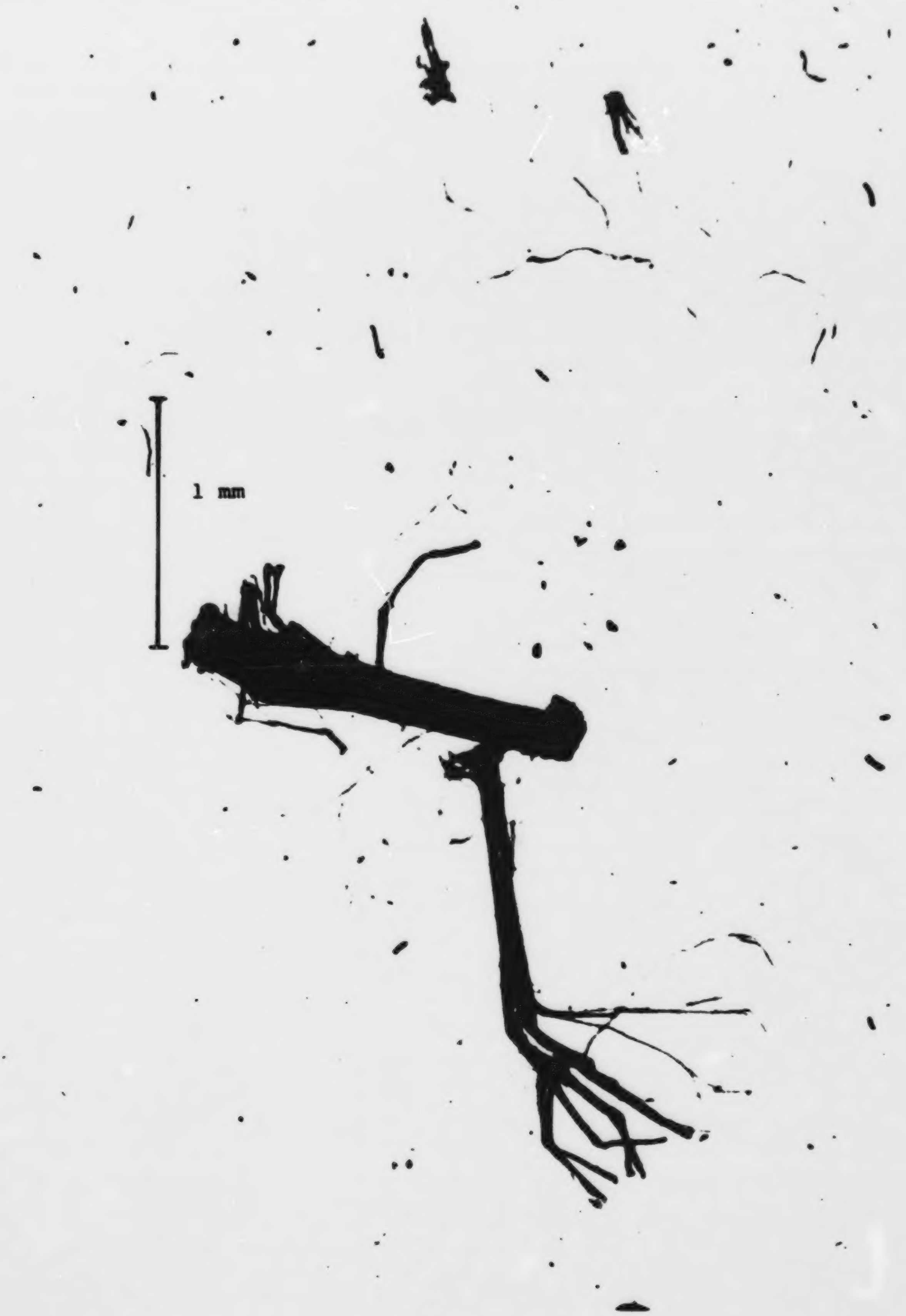


FIG. VII.3i) Cassiar dry-milled AY fibers;
filter loading: 100 μg/cm^2

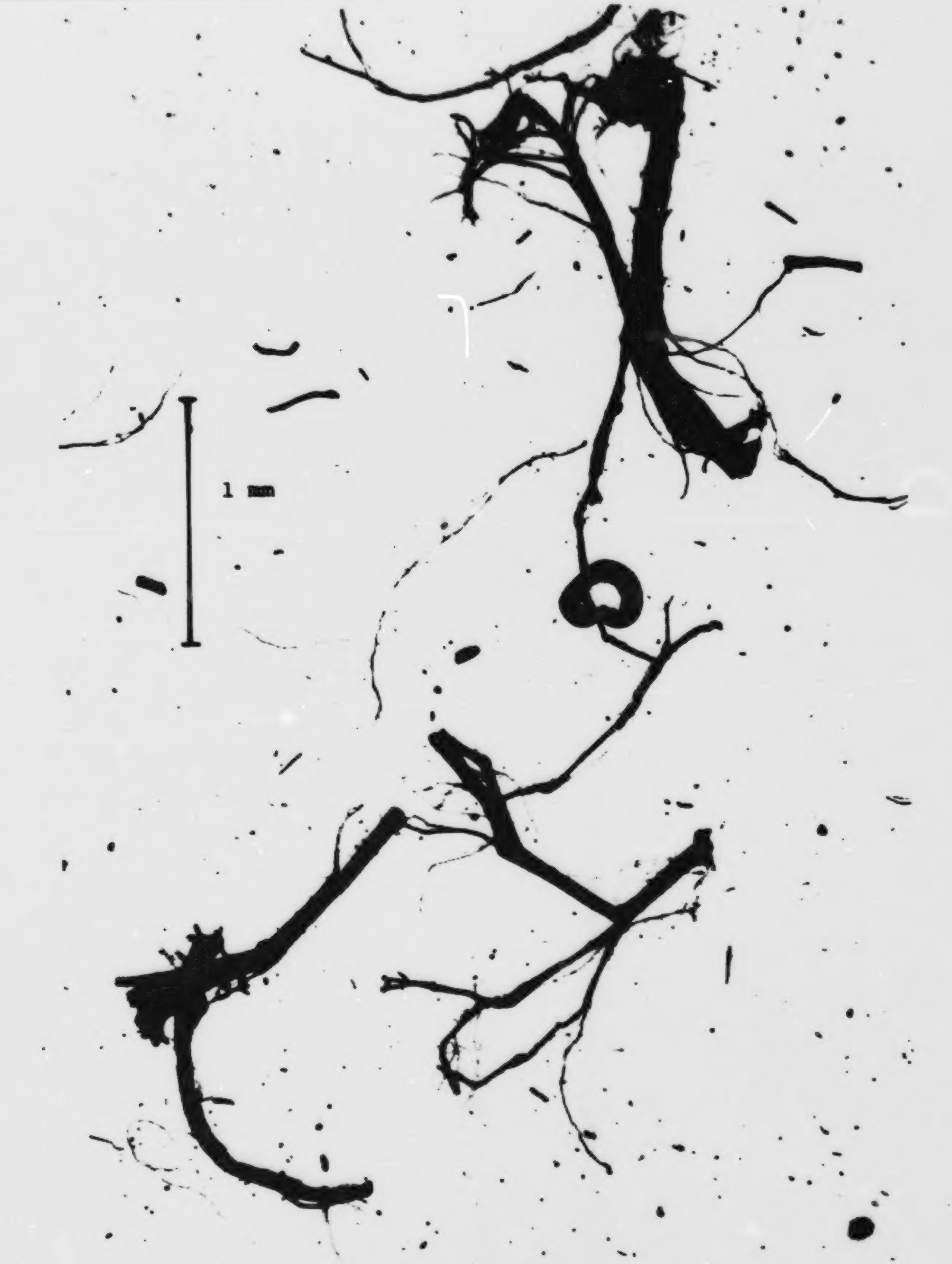


FIG. VII.3j) Cassiar dry-milled AX fibers;
filter loading: 100 $\mu g/cm^2$

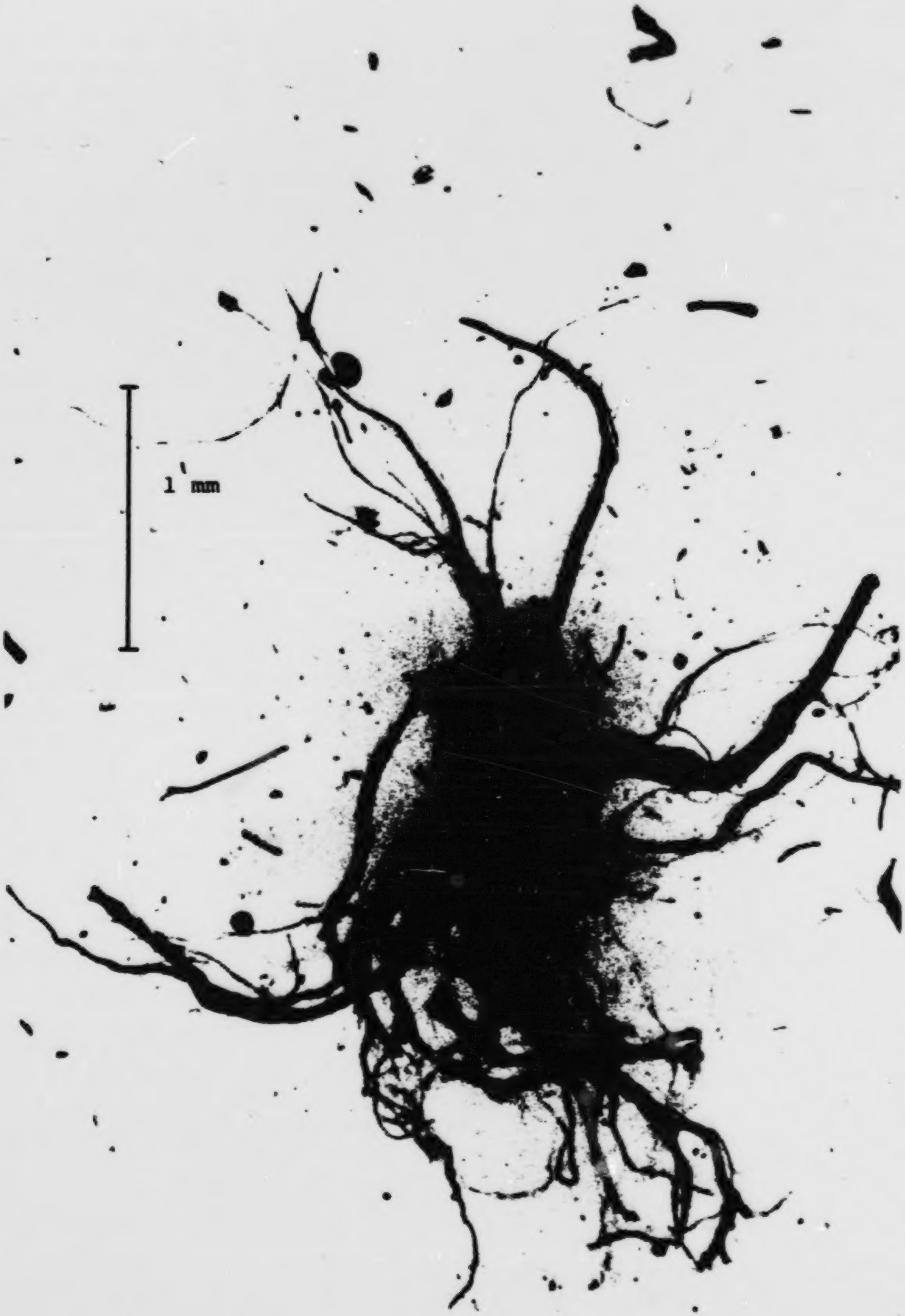


FIG. VII.3k) Quebec dry-milled 4T fibers; filter loading: 100 µg/cm^2

By comparison to the Cassiar fiber products, the wet-milled Woodsreef 10-mesh fibers qualitatively appear intermediate between the wet-milled FEED fibers and the dry-milled AZ, AY and AX products. In particular, the dust-grit content of the Woodsreed product is seen to be higher than for the Cassiar wet-milled products, though lower than for the dry-milled fibers.

On the other hand, no significant morphological distinctions can be established at this stage of our analysis between the dry-milled Cassiar fibers and the Quebec 4T products.

VII.2 - Qualitative features of fibers following Pallmann opening

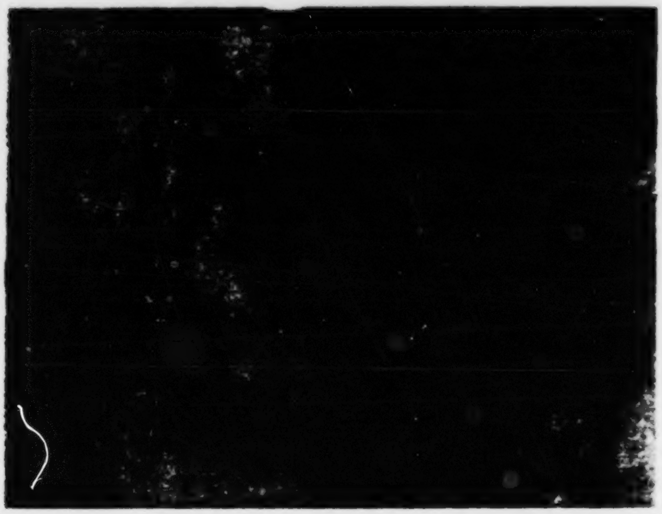
VII.2.a - Optical microscopy observations

We illustrate in Fig. VII.4a and VII.4b, the main qualitative observations noted following fiber opening through a Pallmann mill at 6500 rpm (as for FSU tests) or 13000 rpm. Under the conditions of our experiments (conditions which appeared satisfactory in most strength unit tests), we found that Pallmann opening lead only to a limited separation of the fiber bundles. The Pallmann opening procedure did reduce the number of large bundles, but its major effects on the fibers were found to be (Fig. VII.4):

- the introduction of bends and sharp kinks in the fibers;

- the extensive partial splitting of fibers leading to "broom-" or "tree-type" structures.

a) 6500 rpm



b) 13000 rpm

FIG. VII.4 S fibers opened in a Pallmann mill at different velocities (magnification: 100X; bar: 0.2 mm)

These observations apply to all grades of fibers examined as may be seen from Fig. VII.5a-k. For example, examination of the wet-milled fibers in the series S, M, L and XL reveals, in all cases, an increased "kinking" of the fibers and the frequent occurrence of broom- or tree-type structures. These peculiar structures appear more pronounced in the large bundles, which, of course, are likely to undergo greatest shear in the Pallmann mill. Similar extensive kinking and partial fiber opening is also observed with all dry-milled fiber samples regardless of origin (the kinking effect is somewhat less apparent in the dry-milled fiber products).

VII.2.b - Electron microscopy observations

We reproduce in Fig. VII.6a-c, SEM frames obtained for the CASSIAR S, L and AY fibers after Pallmann opening (6500 rpm). Under these high magnifications, the consequences of Pallmann opening are not as readily apparent as with OM, i.e., Pallmann opening mostly affects the larger fibers which are best seen at lower magnification. Examination of a number of SEM frames suggests the following, in agreement with effects noted above:

- an enhanced frequency of occurrence of sharp bends and kinks;
- an apparent "loosening" of fiber packing or bonding in the larger bundles, most evident with wet-milled S and L fibers.

FIG. VII.5 (following pages)

Optical microscopy (OM) photographs taken from various fiber products following Pallmann opening at 6500 rpm. The approximate surface loading of the filter used is given in $\mu g/cm^2$.

Magnification: 47X; bar: 1 mm

a) Cassiar wet-milled S fibers
b) " " " M "
c) " " " L "
d) " " " XL "
e) " " " FEED "
f) " " " FEED "
g) Woodsreef 10-mesh
h) Cassiar dry-milled AZ fibers
i) " " " AY "
j) " " " AX "
k) Quebec dry-milled 4T fibers

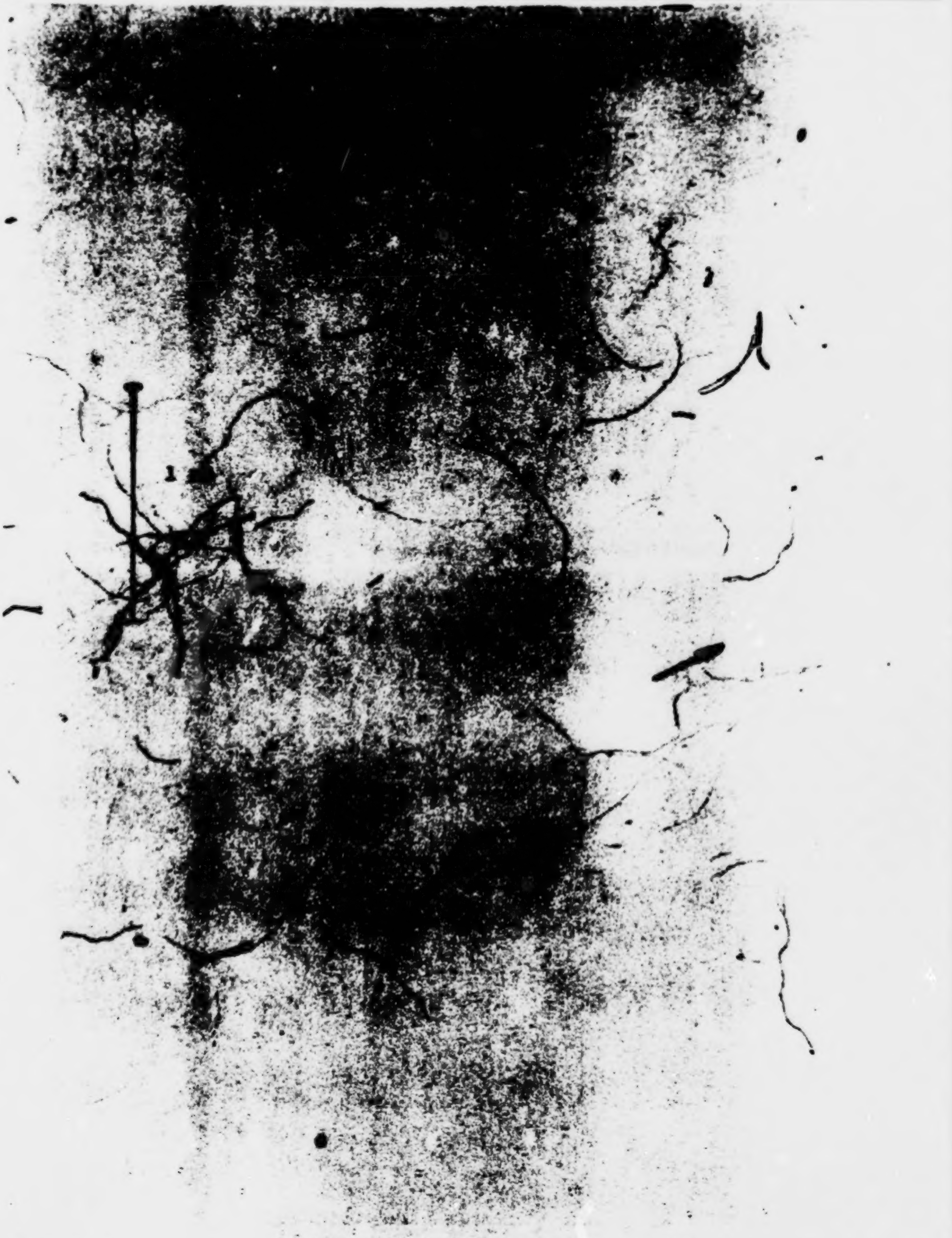


FIG. VII.5a) Cassiar wet-milled S fibers following Pallmann opening at 6500 rpm; filter loading: 25 μg/cm^2

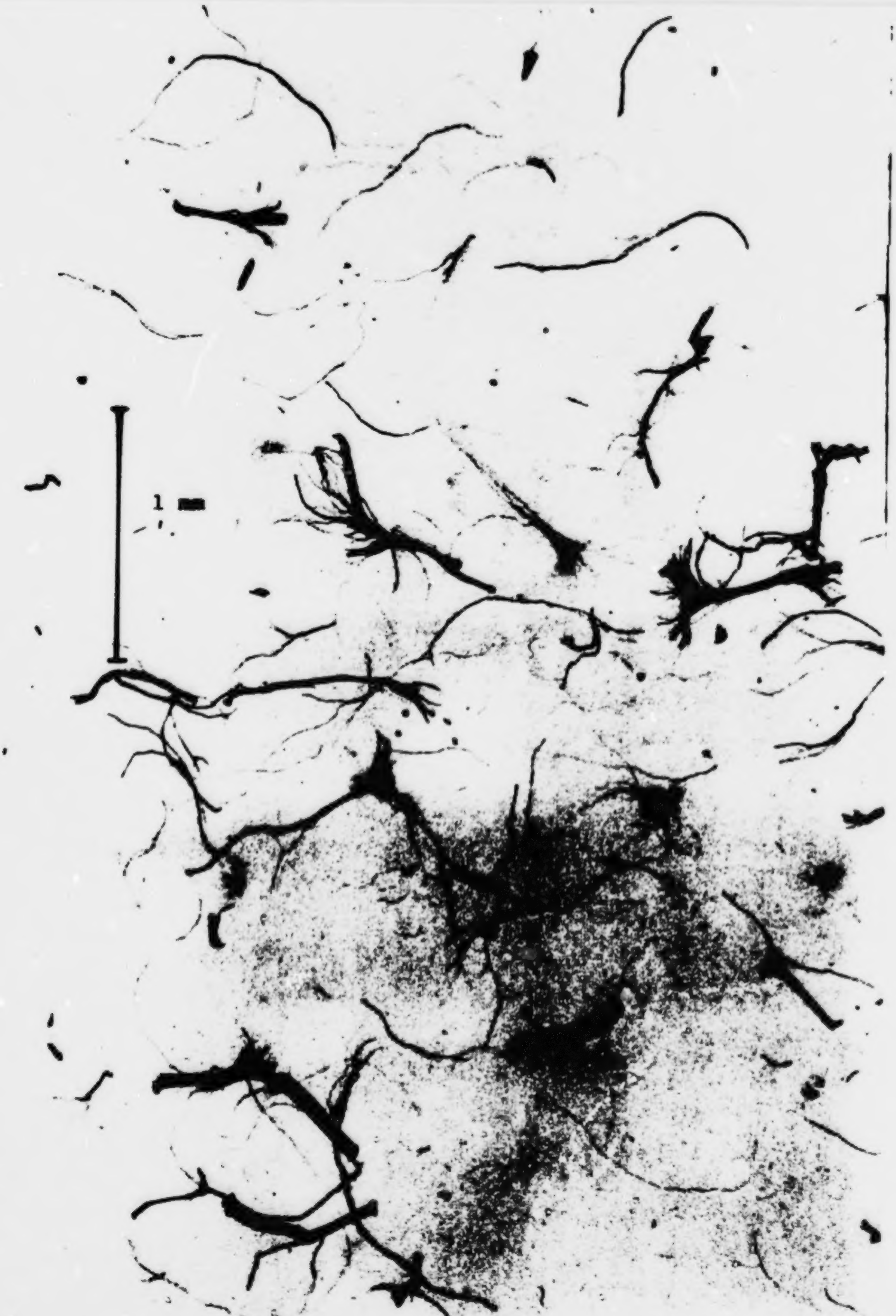


FIG. VII.5b) Cassiar wet-milled M fibers following Pallmann opening at 6500 rpm; filter loading: 50 μg/cm^2

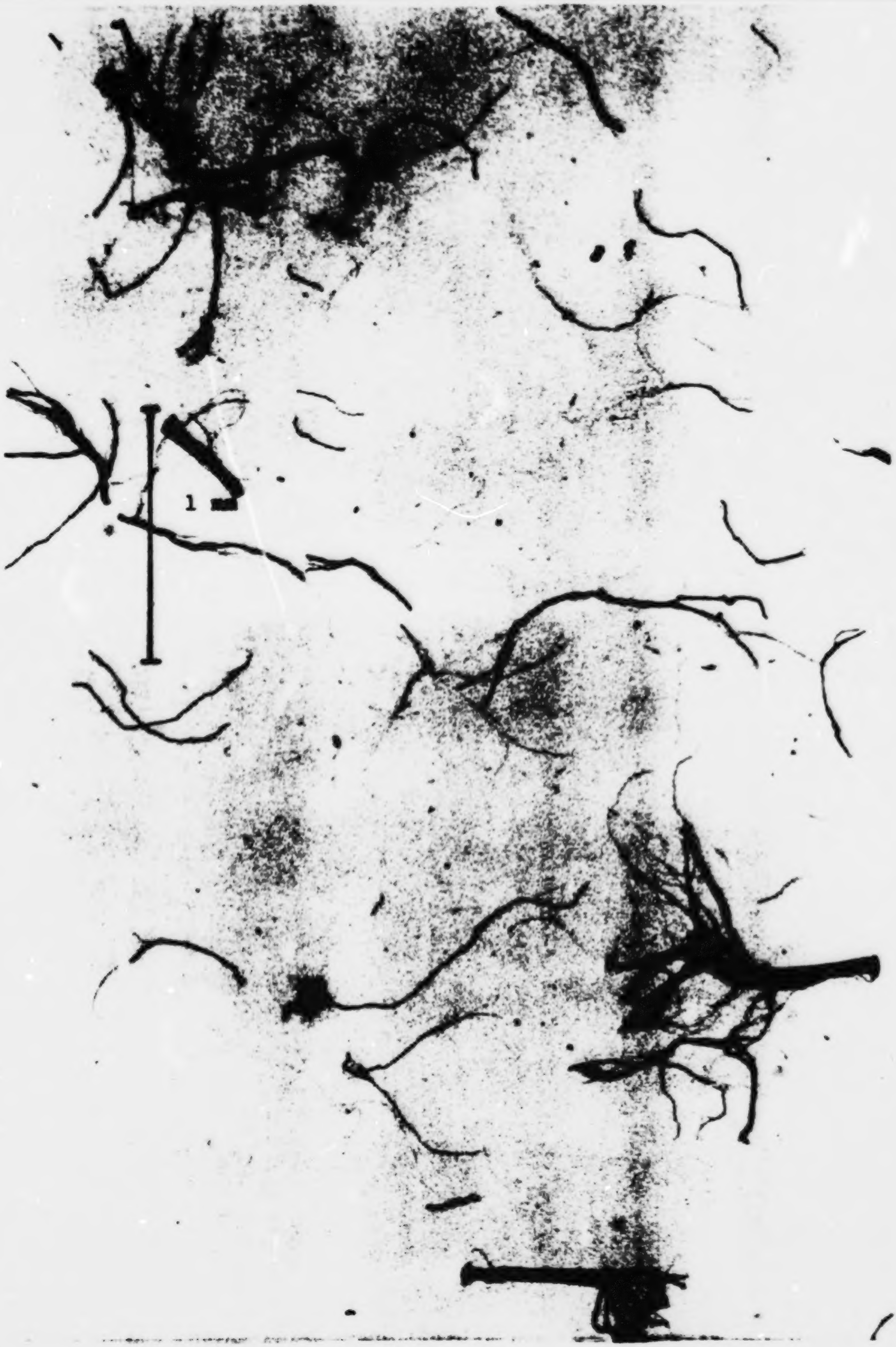


FIG. VII.5c) Cassiar wet-milled L fibers following Pallmann opening at 6500 rpm; filter loading: 50 μg/cm^2

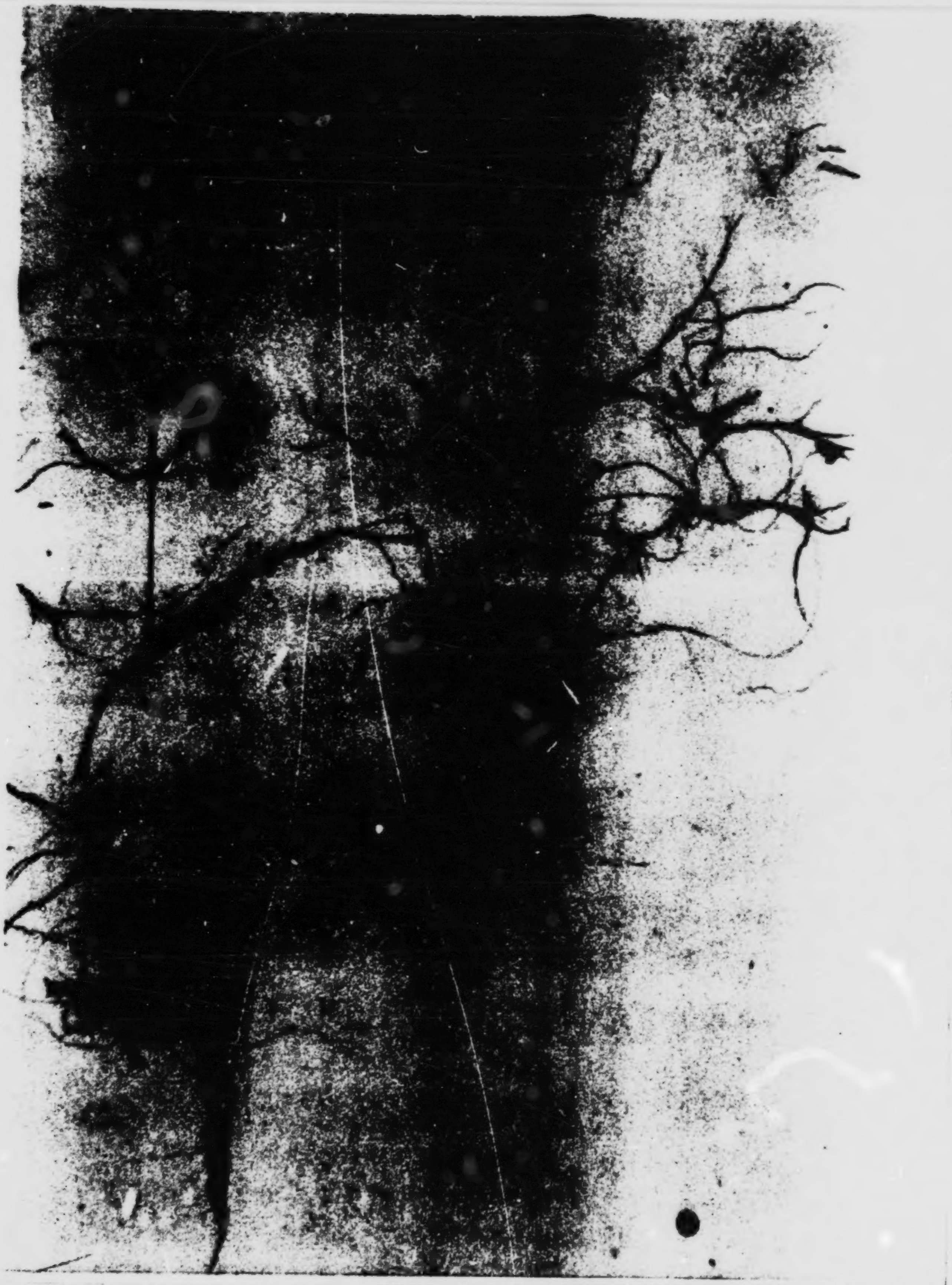


FIG. VII.5d) Cassiar wet-milled XL fibers following Pallmann opening at 6500 rpm; filter loading: 50 µg/cm^2

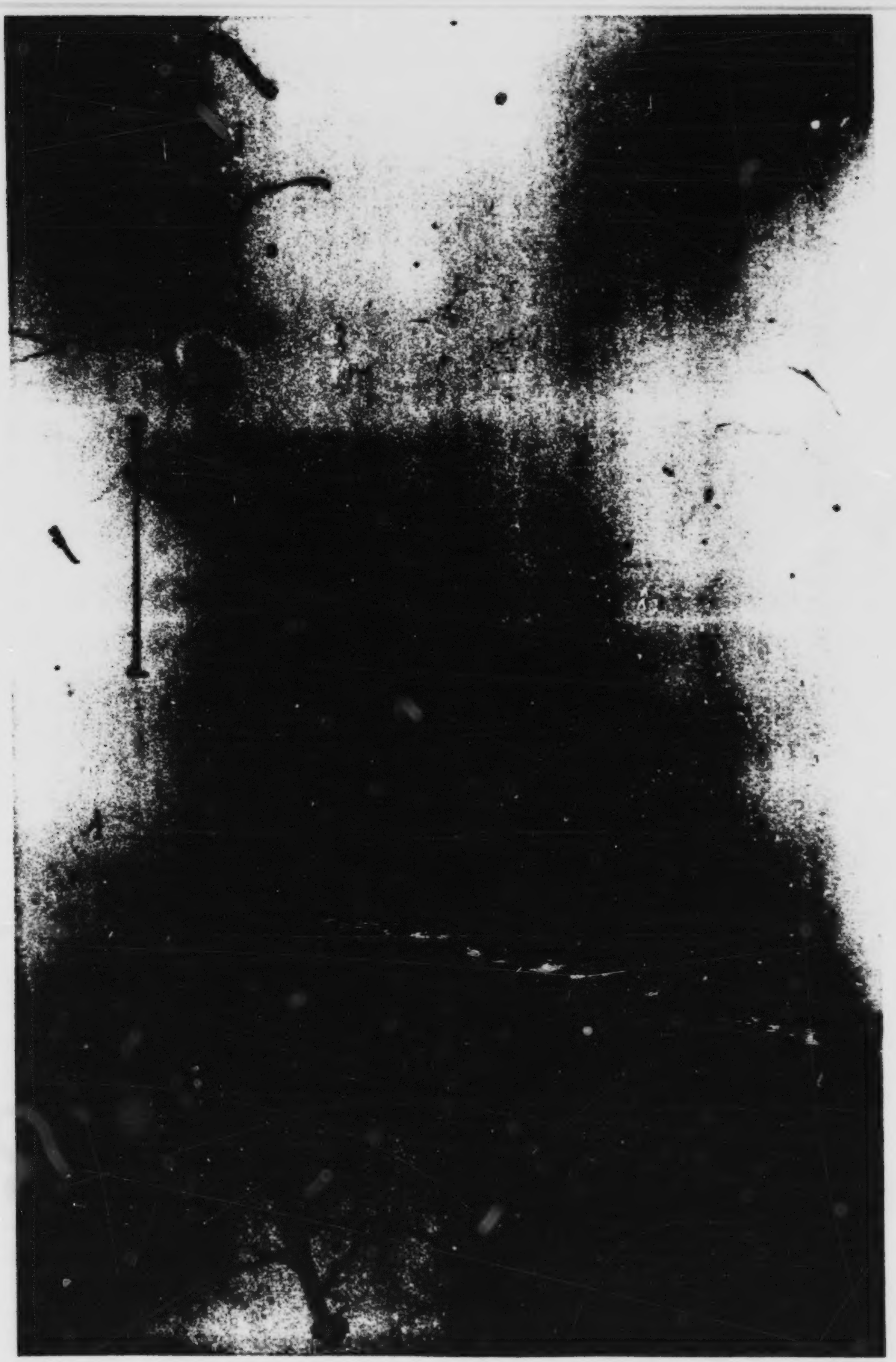


FIG. VII.5e) Cassiar wet-milled FEED fibers following Pallmann opening at 6500 rpm; filter loading: 25 μg/cm^2

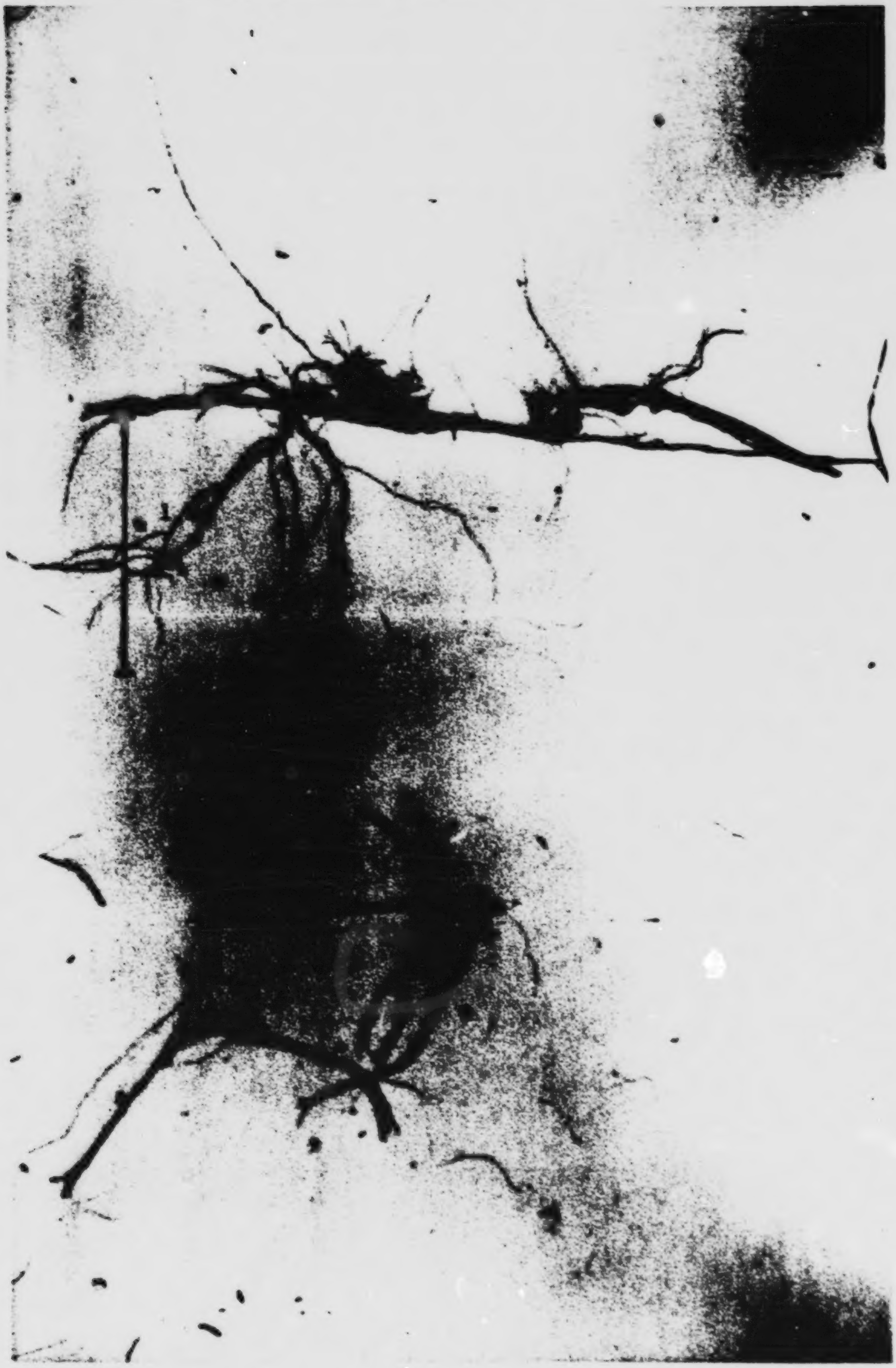


FIG. VII.5f) Same as Fig. VII.5e), different field

FIG. VII.5g) Woodsreef wet-milled 10-mesh fibers following Pallmann opening at 6500 rpm; filter loading: 50 µg/cm^2

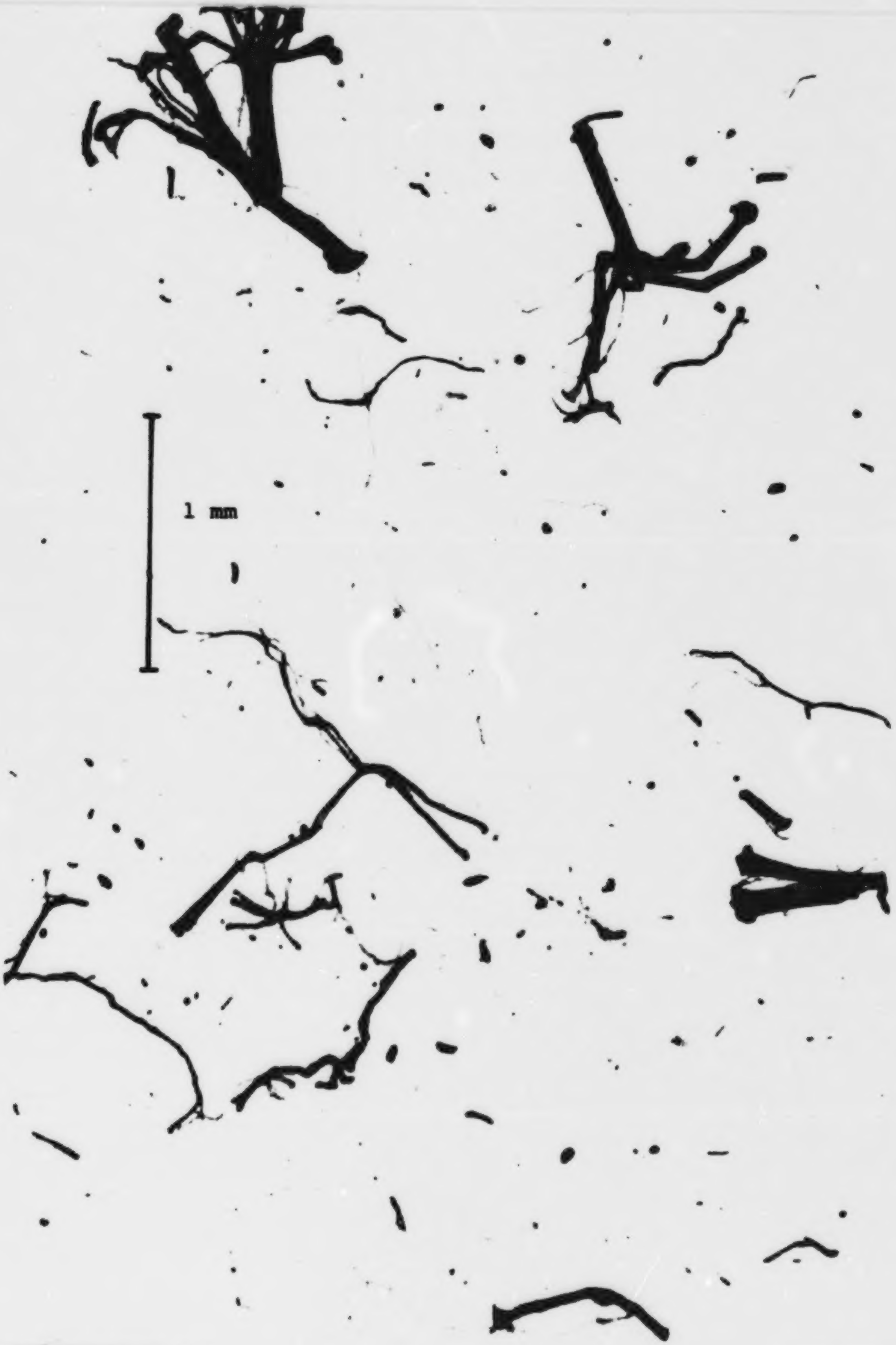


FIG. VII.5h) Cassiar dry-milled AZ fibers following Pallmann opening at 6500 rpm; filter loading: 50 µg/cm^2

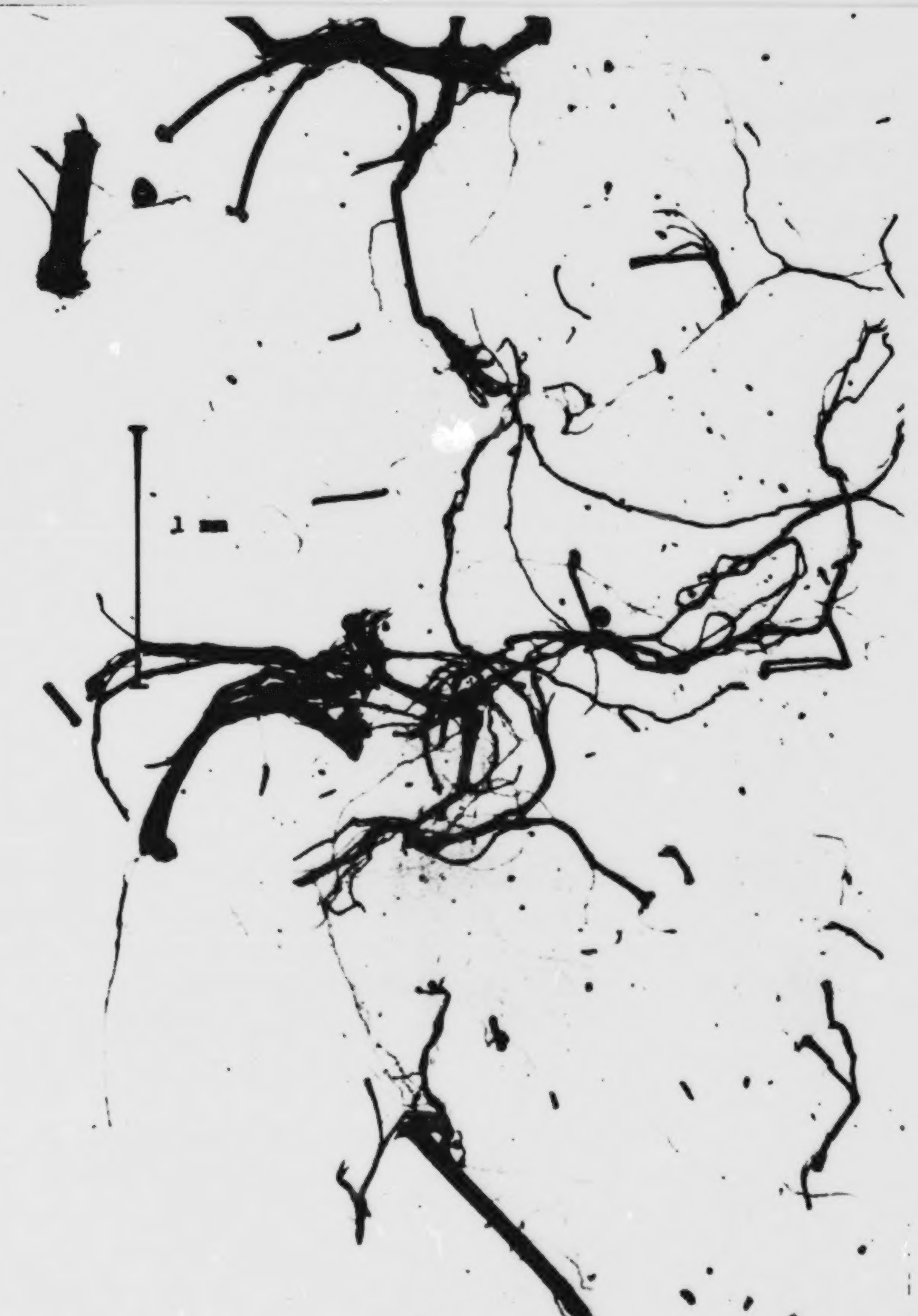


FIG. VII.5i) Cassiar dry-milled AY fibers following Pallmann opening at 6500 rpm; filter loading: 50 μg/cm^2

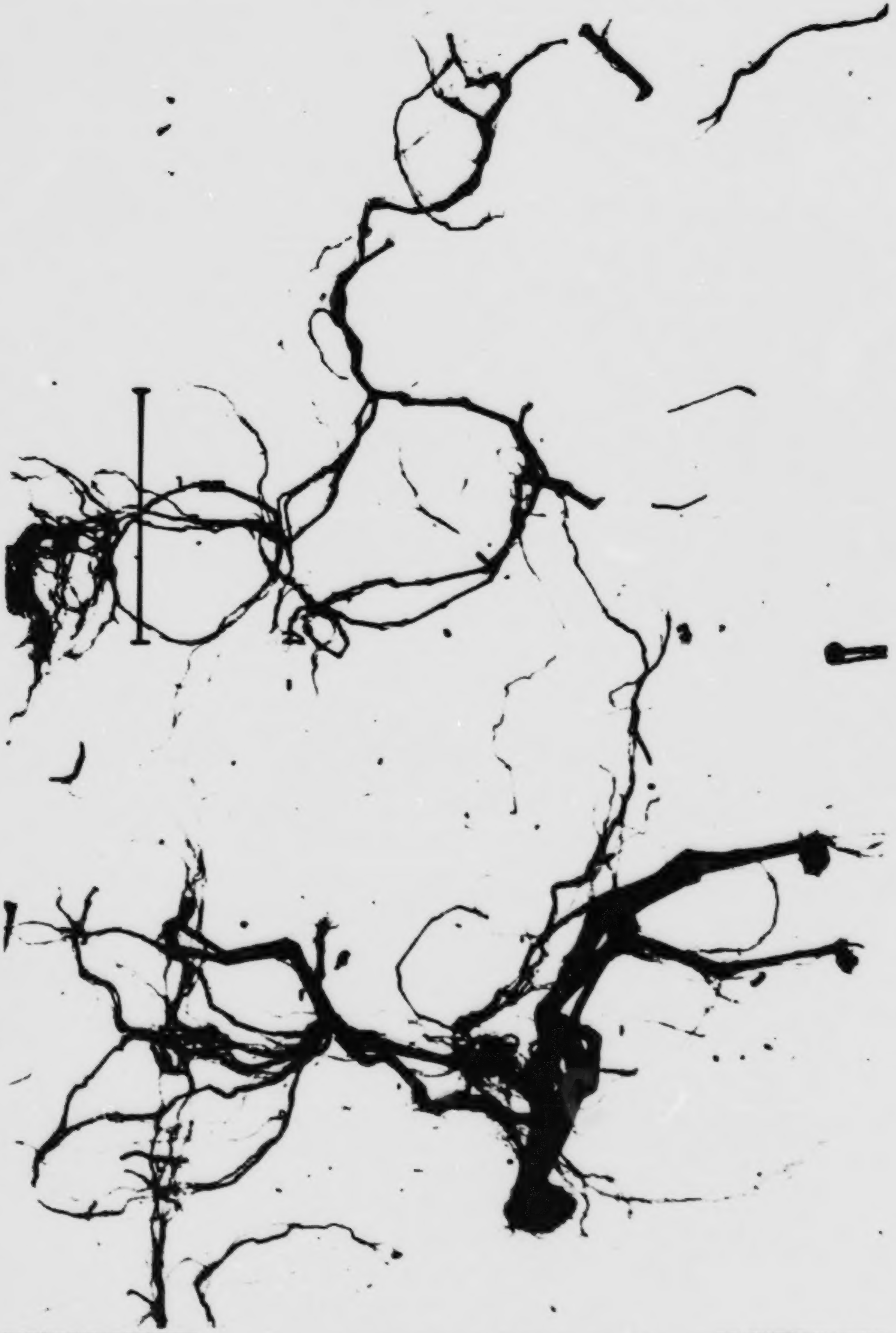


FIG. VII.5j) Cassiar dry-milled AX fibers following Pallmann opening at 6500 rpm; filter loading: 50 µg/cm^2

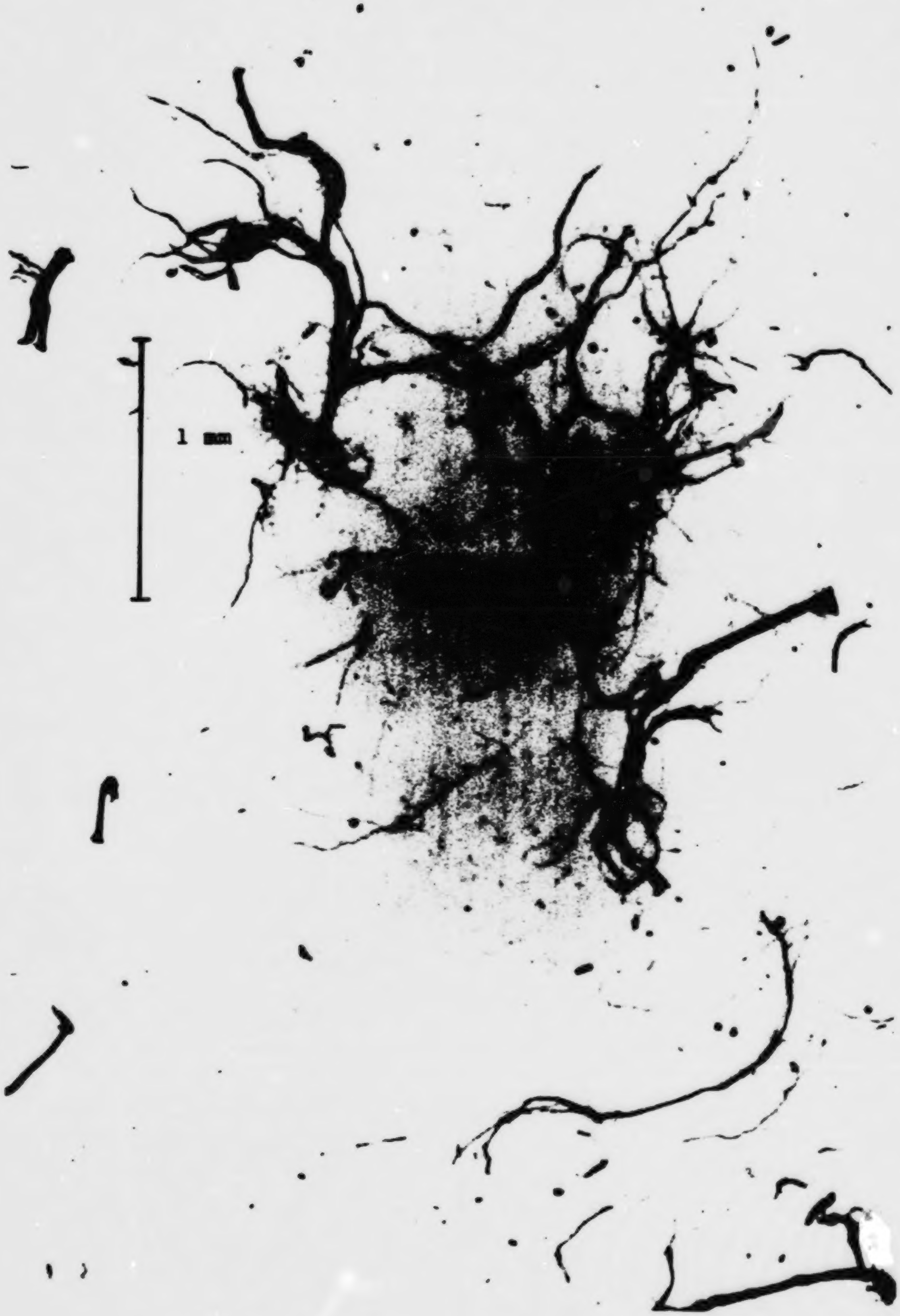


FIG. VII.5k) Quebec dry-milled 4T fibers following Pallmann opening at 6500 rpm; filter loading: 25 μg/cm^2

FIG. VII.6 (following pages)

Scanning electron microscopy (SEM) frames obtained for several wet- and dry-milled Cassiar fibers after Pallmann opening at 6500 rpm.

Upper frame : magnification: ~700X
vertical bar : 100 μm
Lower frame : magnification: 2100X
vertical bar : 10 μm

a) Wet-milled S fibers
b) Wet-milled L fibers
c) Dry-milled AY fibers

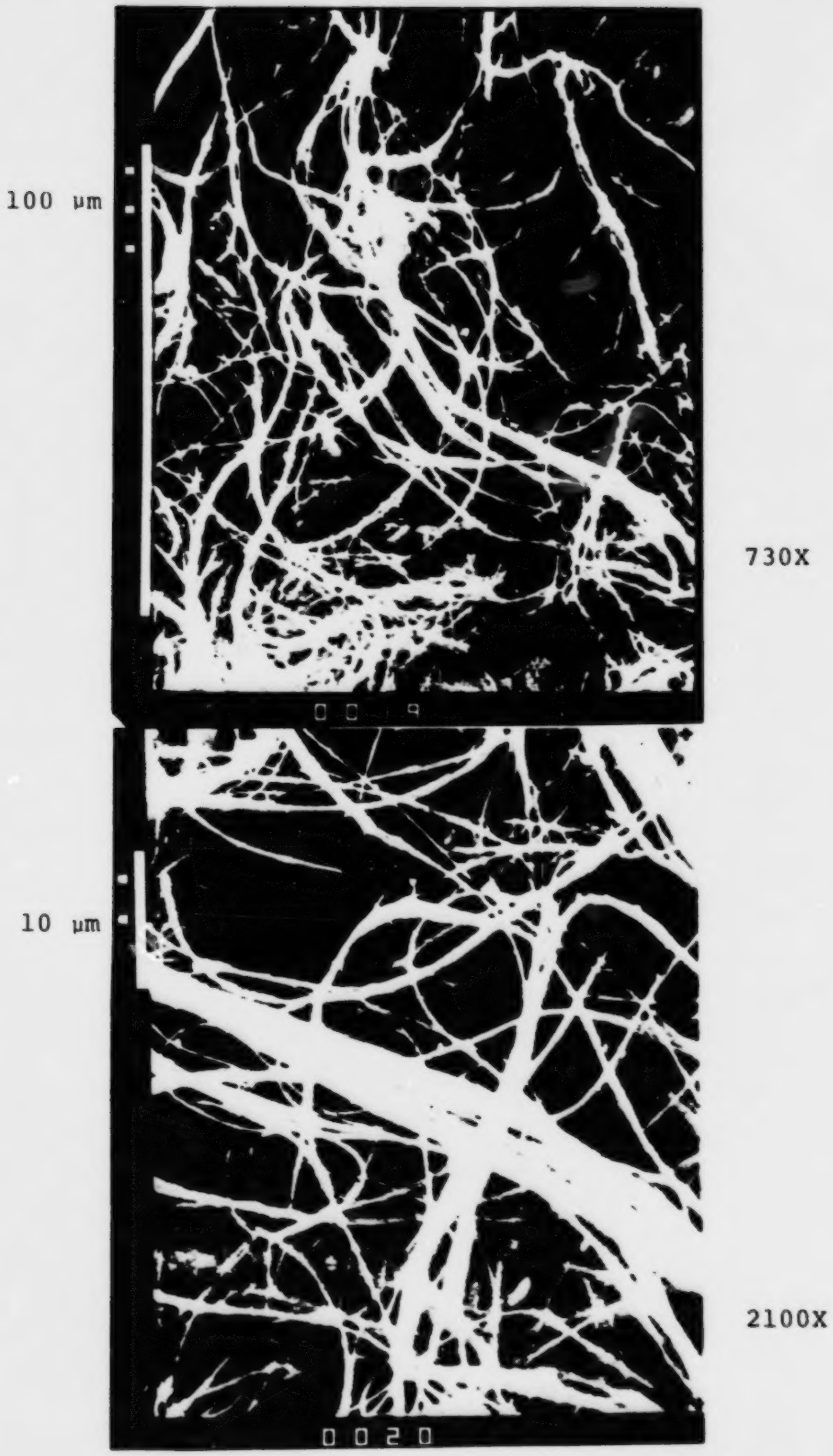

100 µm
730X
10 µm
2100X
0020

FIG. VII.6a) Cassiar wet-milled S fibers after Pallmann opening at 6500 rpm

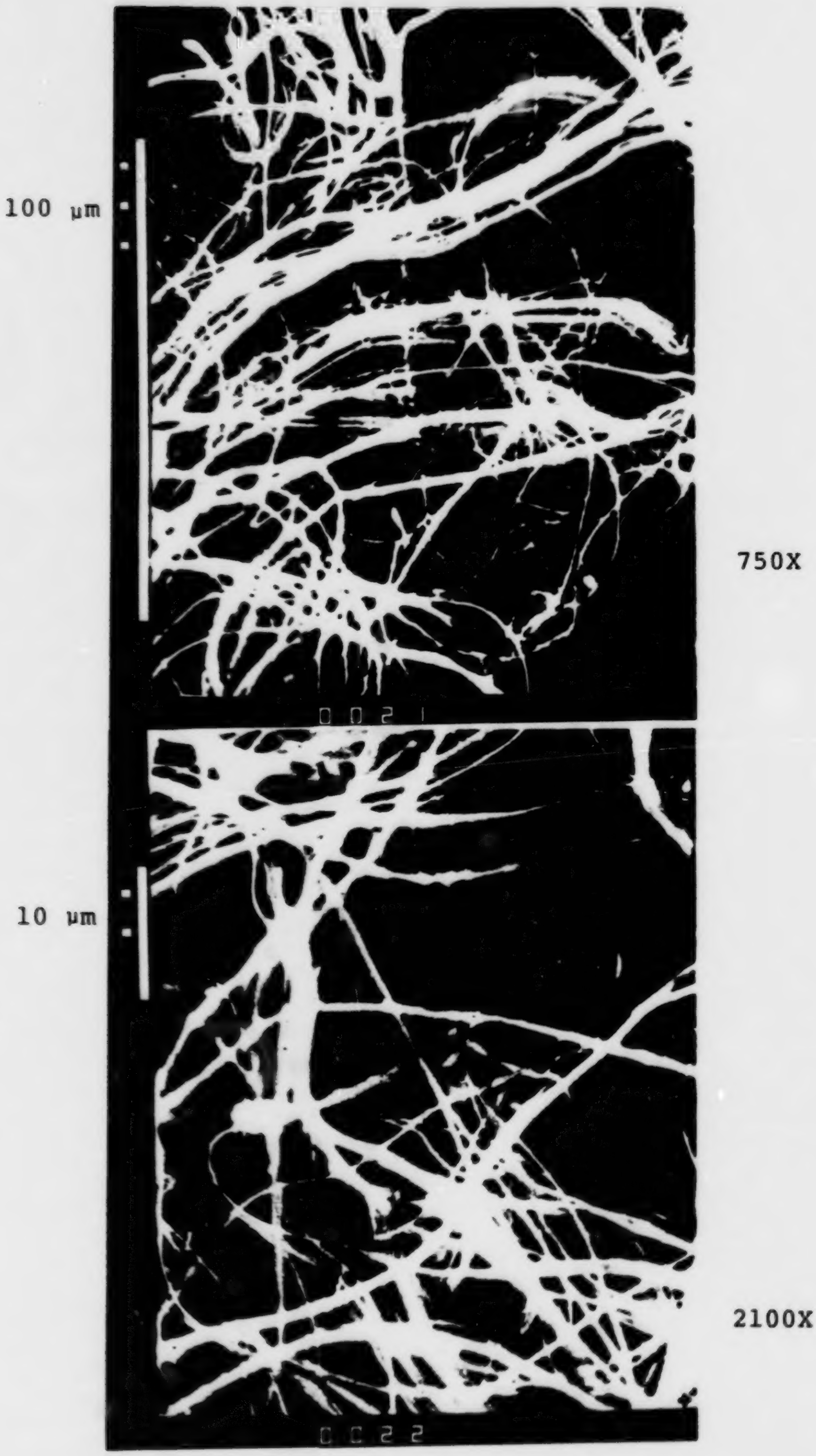

100 µm
750X
0021
10 µm
2100X
0022

FIG. VII.6b) Cassiar wet-milled L fibers after Pallmann opening at 6500 rpm

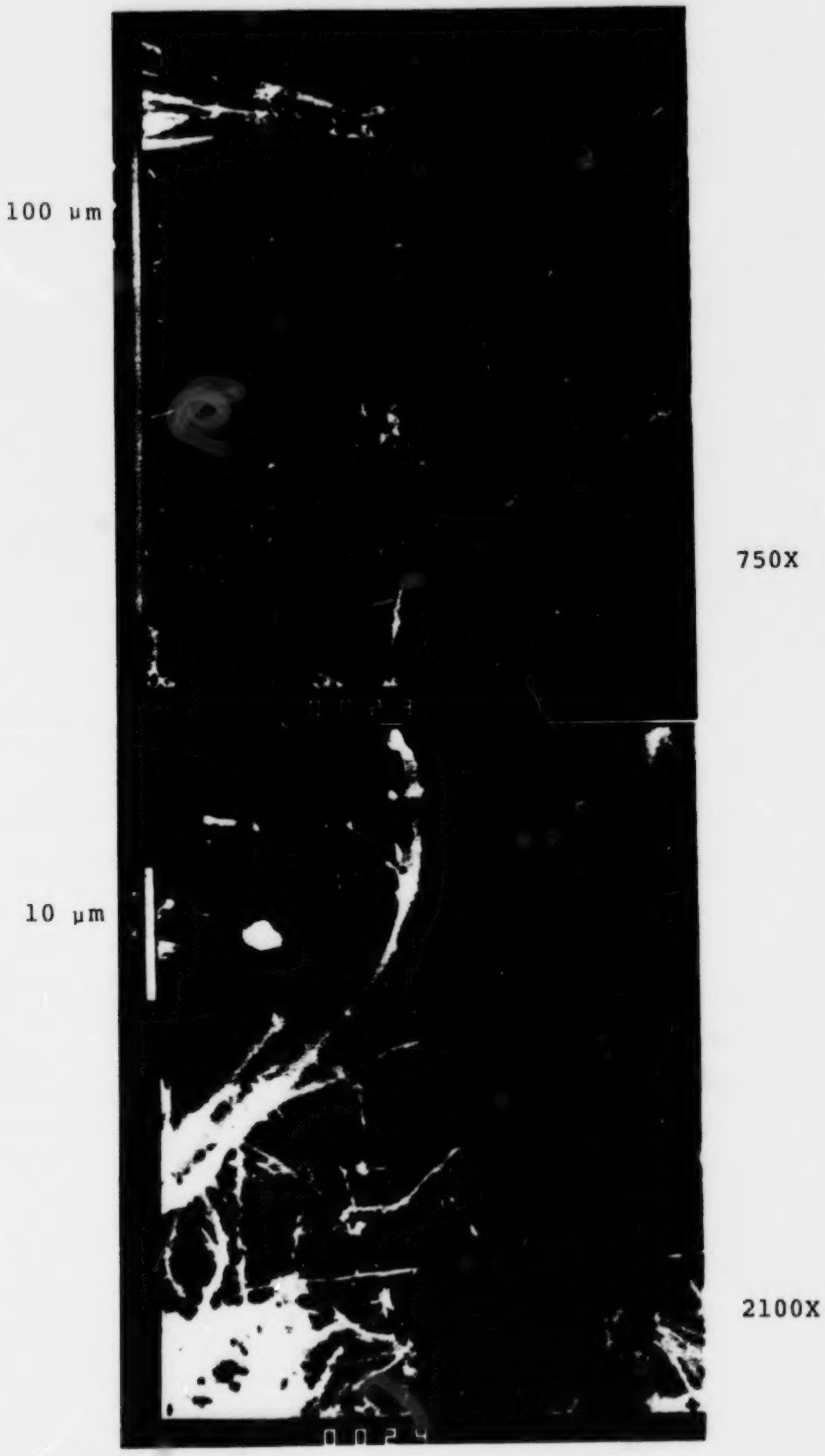

100 µm
750X
10 µm
2100X

FIG. VII.6c) Cassiar dry-milled AY fibers following Pallmann opening at 6500 rpm

VII.3 Qualitative features of fibers opened via alternative procedures

Various alternative procedures of fiber opening were attempted to enhance the degree of opening with minimal fiber breaking. Ultrasonic probes frequently used to disperse fiber slurries were found to induce considerable fiber length reduction at high dispersion energies. Among the more "gentle" fiber opening methods, the wet turbine and the Dounce tube were found most interesting.

The aqueous turbine used in these fiber opening experiments is basically a stirred container with fixed baffles and an impeller stirrer having a specific design. This equipment was made available at Ceram-sna, Sherbrooke and is described in Appendix F.

The Dounce tube is a simple device consisting of a high precision bore glass tube through which a glass sphere of closely fitting diameter is displaced up and down. The dilute aqueous fiber slurry is forced through the narrow space between the sphere and the tube wall, leading to high longitudinal shear.

We illustrate in Fig. VII.7a-e typical frames (47X) of several fiber samples opened through the alternative turbine or Dounce tube methods. With both fiber opening techniques, the resulting fibers exhibit fewer kinks and broom- or tree-type structures than those obtained through Pallmann opening. The turbine-opened fibers, however, still contain a significant amount of thick compact fiber bundles, presumably because the shearing action in the turbine is too weak. On the other hand, the Dounce tube method yields highly opened fibers; to date, this appears the "best" opening method to yield homogeneous fibers with minimal kinks or breaks. Dounce tube opening is currently a small scale laboratory method, but its principle is the same as that of a Szego mill.

FIG. VII.7 (following pages)

Optical microscopy (OM) frames for several types of fibers opened using a high speed aqueous turbine or a Dounce tube. The approximate surface loading of the filter used is given in µg/cm^2

a) Cassiar S fibers, turbine opened
b) " M " " "
c) " L " " "
d) " XL " " "
e) Quebec 4T, Dounce opened

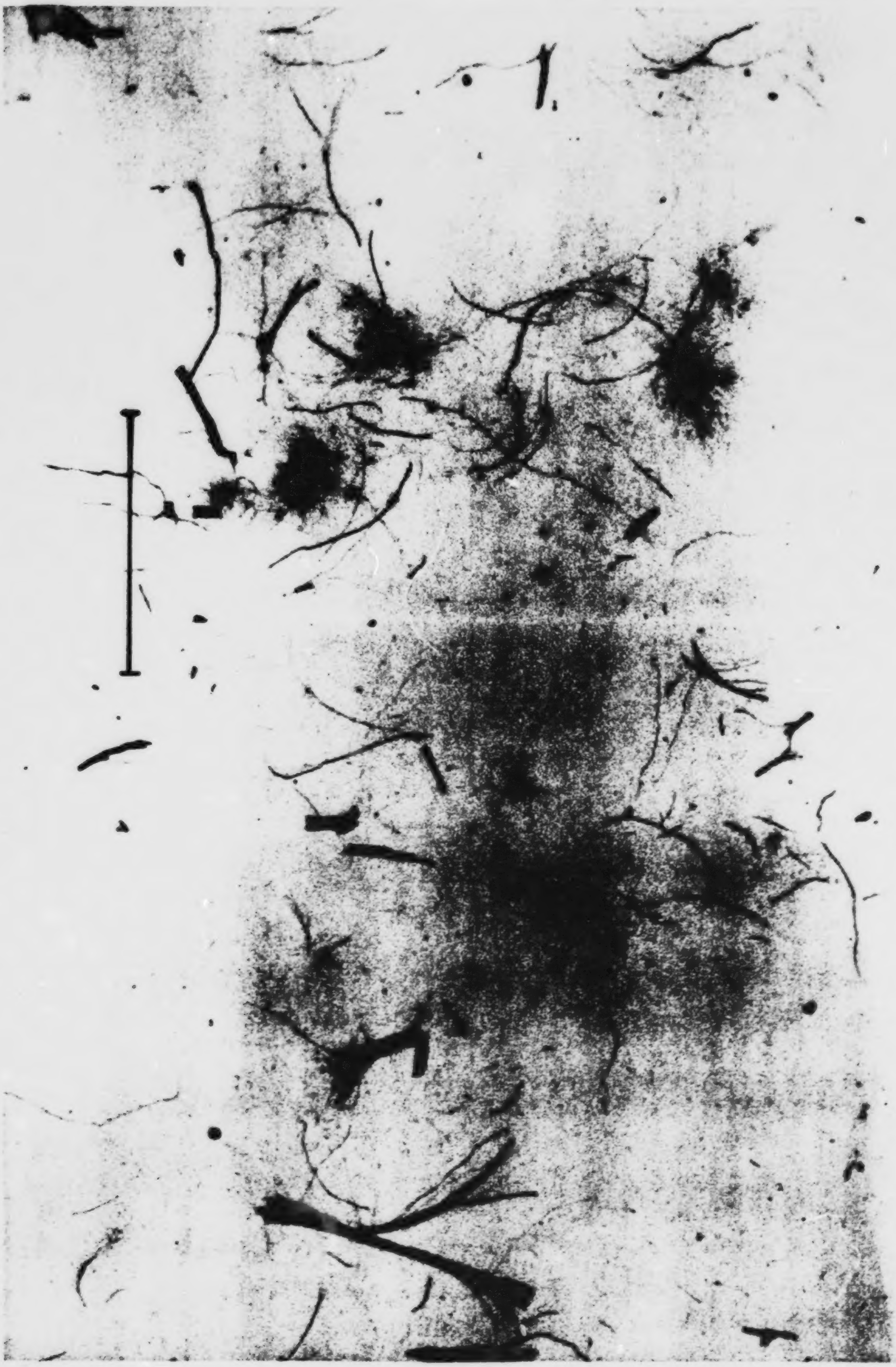


FIG. VII.7a) Cassiar wet-milled S fibers opened through a wet turbine; filter loading: 25 µg/cm^2

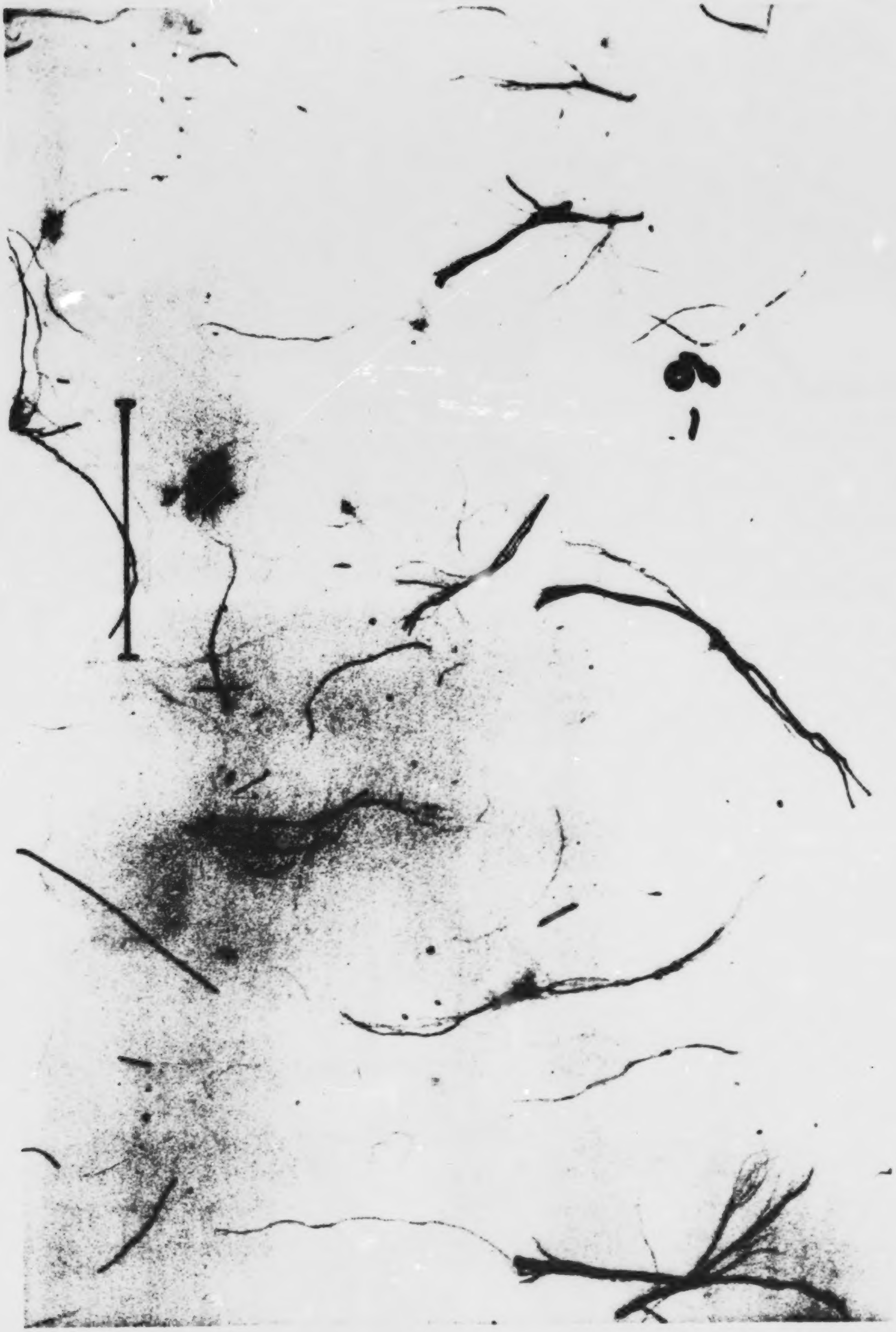


FIG. VII.7b) Cassiar wet-milled M fibers opened through a wet turbine; filter loading: 25 µg/cm^2

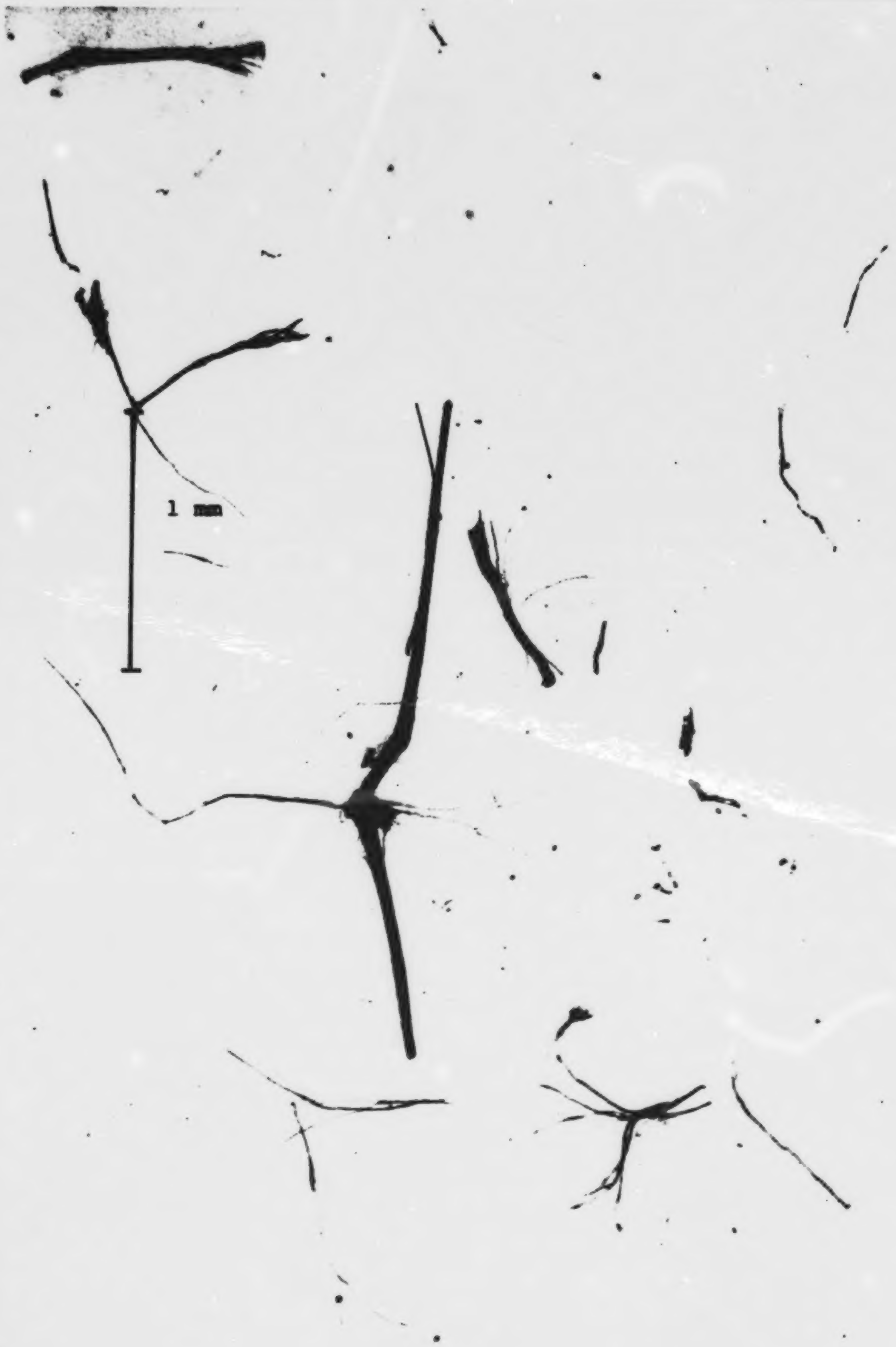


FIG. VII.7c) Cassiar wet-milled L fibers opened through a wet turbine; filter loading: 50 μg/cm²

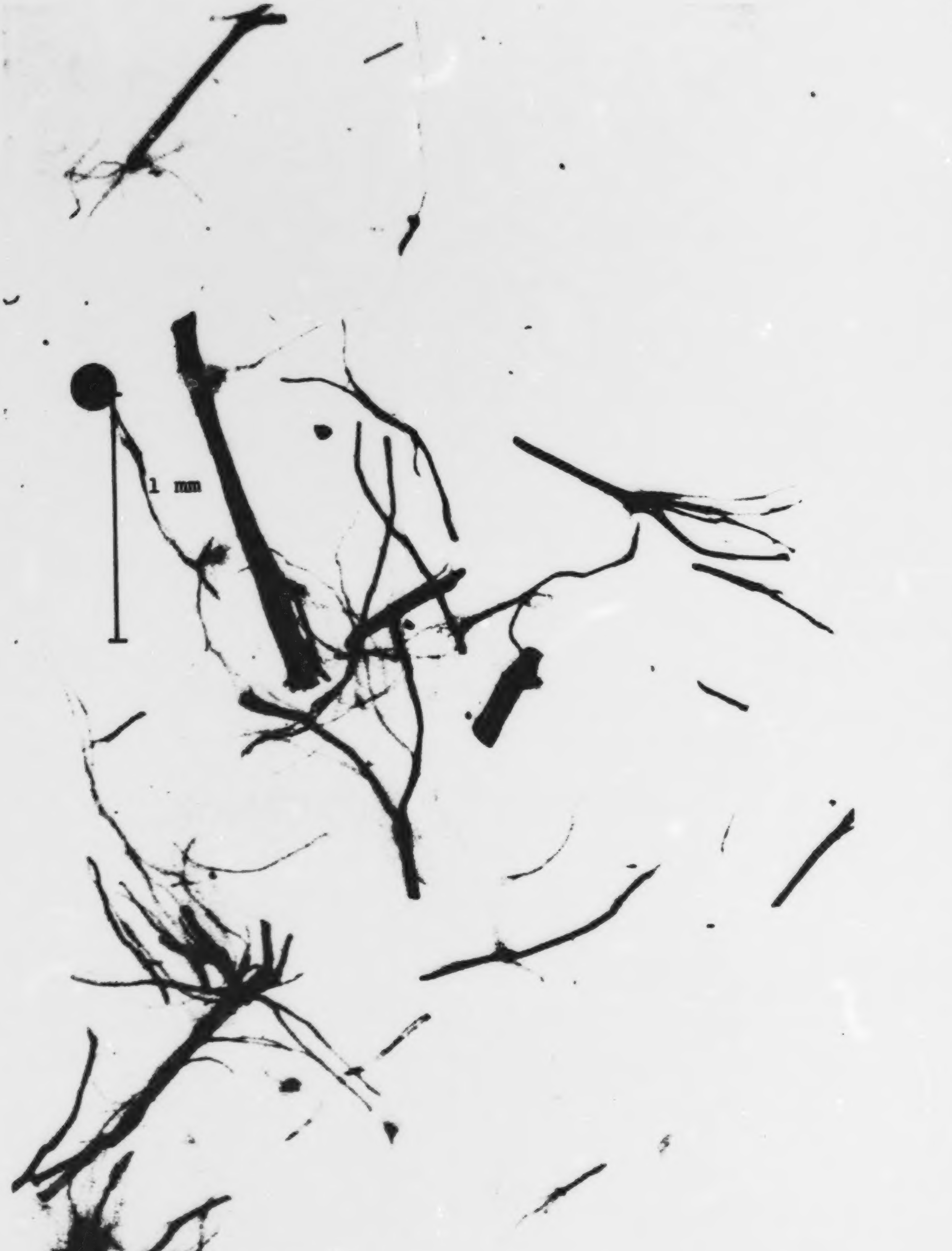


FIG. VII.7d) Cassiar wet-milled XL fibers opened through a wet turbine; filter loading: 50 μg/cm^2

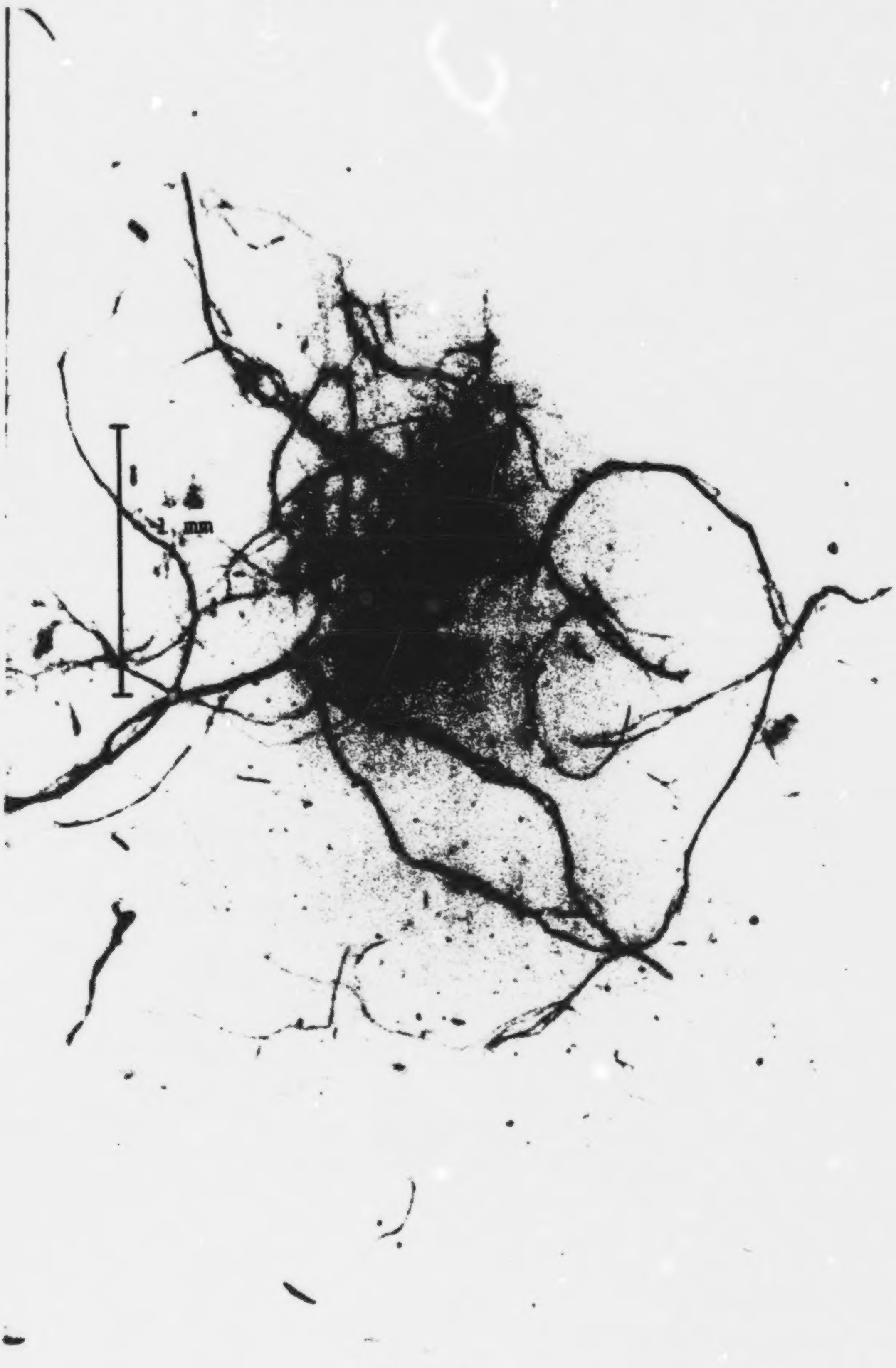


FIG. VII.7e) Quebec dry-milled 4T fibers opened through a Dounce device; filter loading: 50 μg/cm^2

VII.4 - Summary of key observations

OM and SEM examination of bulk and Pallmann opened wet- and dry-milled fibers lead to the following qualitative conclusions:

- Wet-milled fibers are systematically found to contain fewer non fibrous (dust, grit) particles than the corresponding dry-milled fibers.
- The overall appearance of wet-milled fibers shows fewer thick compact bundles in these materials compared to dry-milled products; the fiber bundles found in wet-milled samples frequently exhibit a "loose bundle" (loosely bonded) appearance.
- The S, M, L, XL subgroups obtained from the bulk wet-milled fibers (FEED) exhibit, from S to XL: 1) increasing average lengths and diameters and 2) decreasing size homogeneity (larger bundles are increasingly present from S to XL).
- Pallmann mill opening of the various fibers examined (wet-milled: Cassiar S, M, L, XL and Woodsreef 10-mesh dry-milled: Cassiar AX, AY, AZ and Quebec 4T) greatly reduces the number of large compact bundles in the sample; Pallmann opening further leads to extensive "kinking" and partial splitting of the fibers leading to "broom-" and "tree-type" structures; for thin fibers, it appears the main effect of Pallmann milling is to introduce "kinks" and sharp bends, presumably leading to some fiber breaking.
- Turbine opening of wet-milled fibers in aqueous slurries is more "gentle" than Pallmann mill opening, but it leaves a substantial amount of thick compact bundles.
- Dounce tube opening in dilute aqueous slurries appears as an ideal (laboratory) method to produce highly opened fibers with minimal kinks and breaks.

Some of the observations noted with regards to fiber opening, especially through the Pallmann mill, may have meaningful consequences in other fiber tests. The "kinking" and "partial splitting" of fibers should enhance the mechanical retention of the fibers in a cement matrix, therefore improving the results of strength tests. Most certainly, the presence of kinks, split ends and tree-type structures will enhance the bulk volume of the opened fibers, a property which is generally found well related to FSU results.

VIII - FIBER SIZE ANALYSIS FROM OPTICAL MICROSCOPY

We report below distributions of fiber lengths (ℓ) and diameters (d) obtained from measurements on optical microscopy photographs taken at 47X magnification (similar to these shown in Fig. VII.3, 5 and 7). The resolution limit of ℓ and d measurements was 2 µm; details of the methodology and statistical analysis are given in Sections V.5 and Appendices A and B.

VIII.1 - Results for bulk fibers

a) Length distributions

The length distributions of the various wet- and dry-milled fiber products are illustrated in Fig. VIII.1a-j. For each type of fibers examined, we report first a fiber population distribution (upper histograms) and a length normalized distribution (lower histograms).

The population distribution is simply a histogram of the number of fibers in consecutive length groups separated by 0.05 mm (50 µm) increments; the mean value obtained from this distribution is the true average length of the sample. The normalized distribution shows the contribution of each length class to the total length of the fibers in the sample; the mean obtained from the normalized distribution represents the average lengths of the fibers which contribute most to the total length of the sample.

CASSIAR WET-MILLED S FIBERS (Bulk)

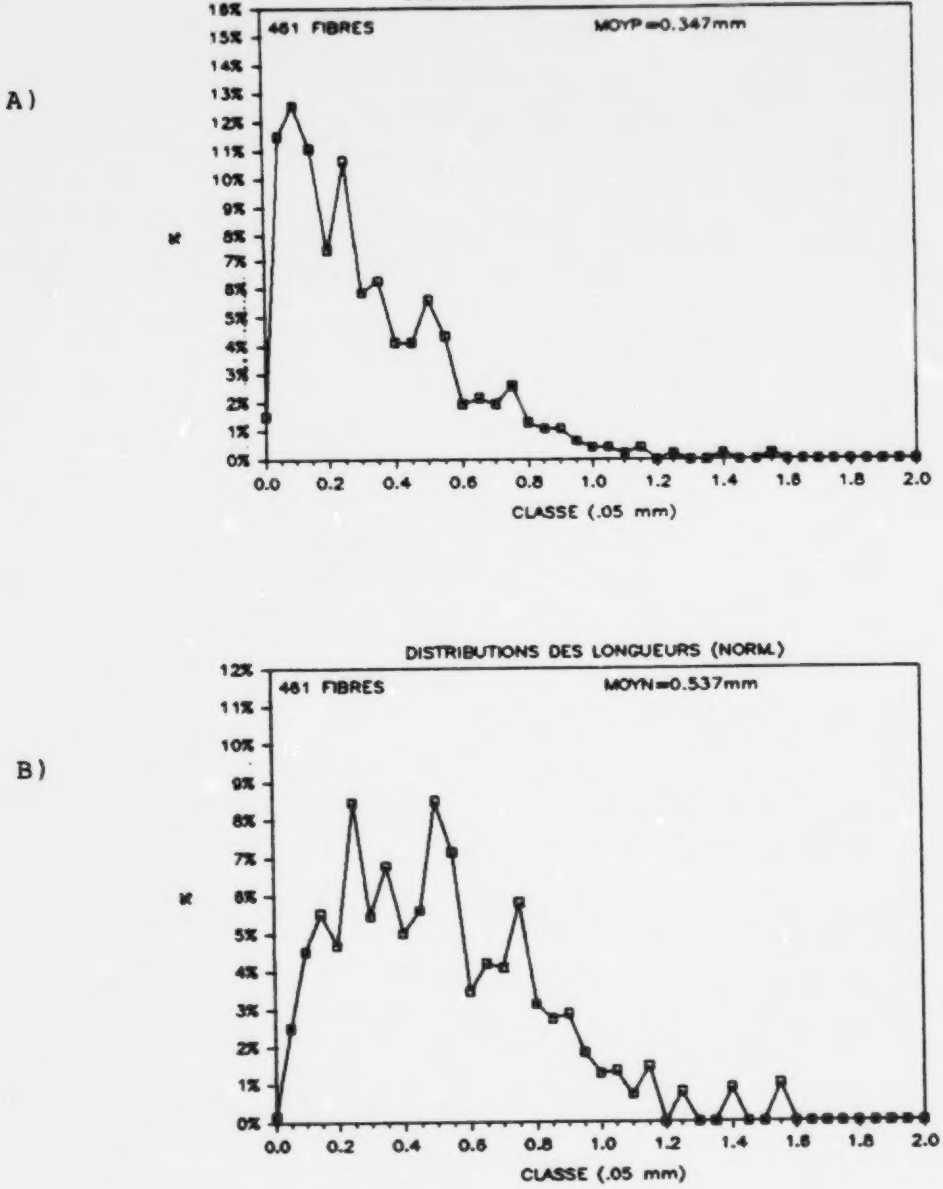


FIG. VIII.1a) Length distributions of S fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

(Class labels on X coordinates give the lower limit of each class.)

CASSIAR WET-MILLED M FIBERS (Bulk)

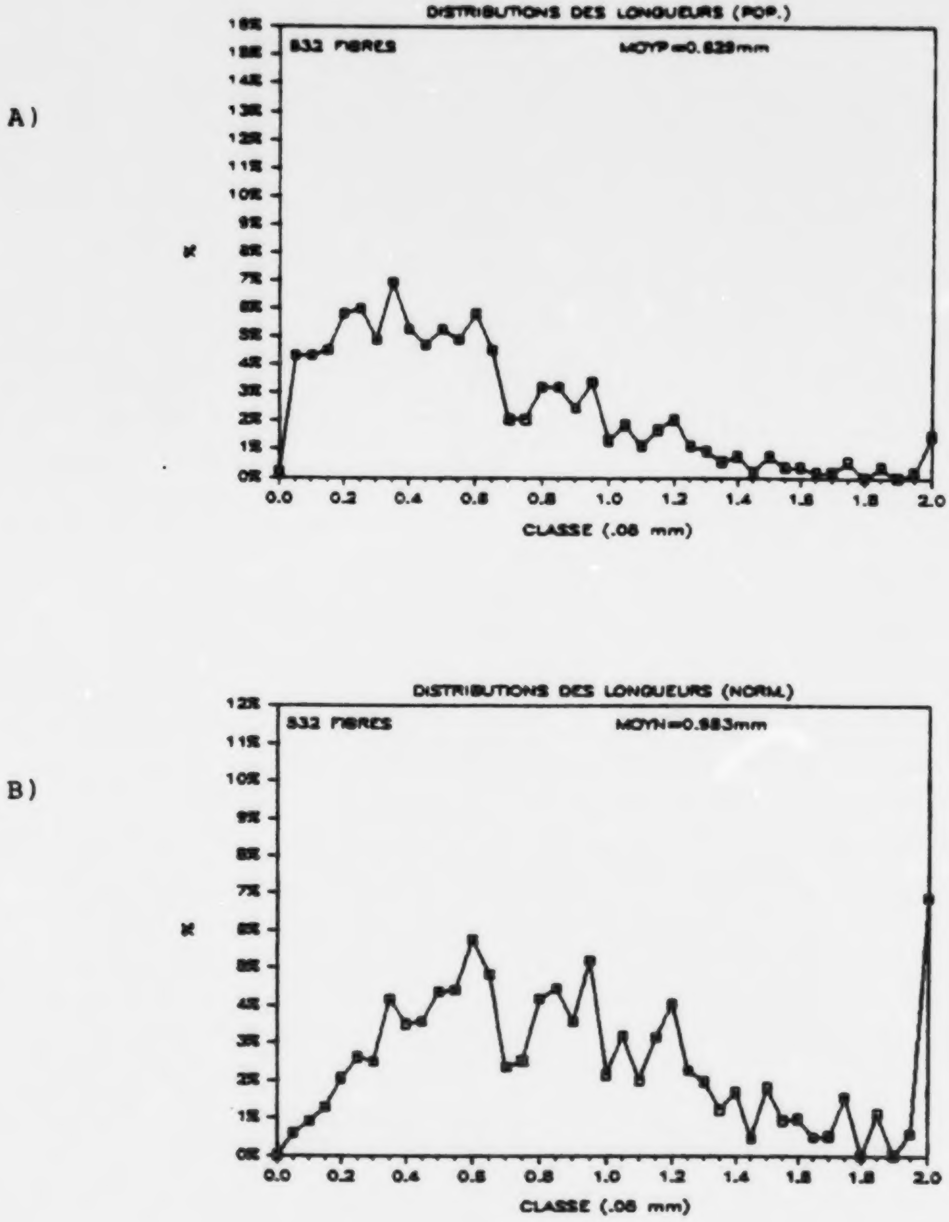


FIG. VIII.1b) Length distributions of M fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

CASSIAR WET-MILLED L FIBERS (Bulk)

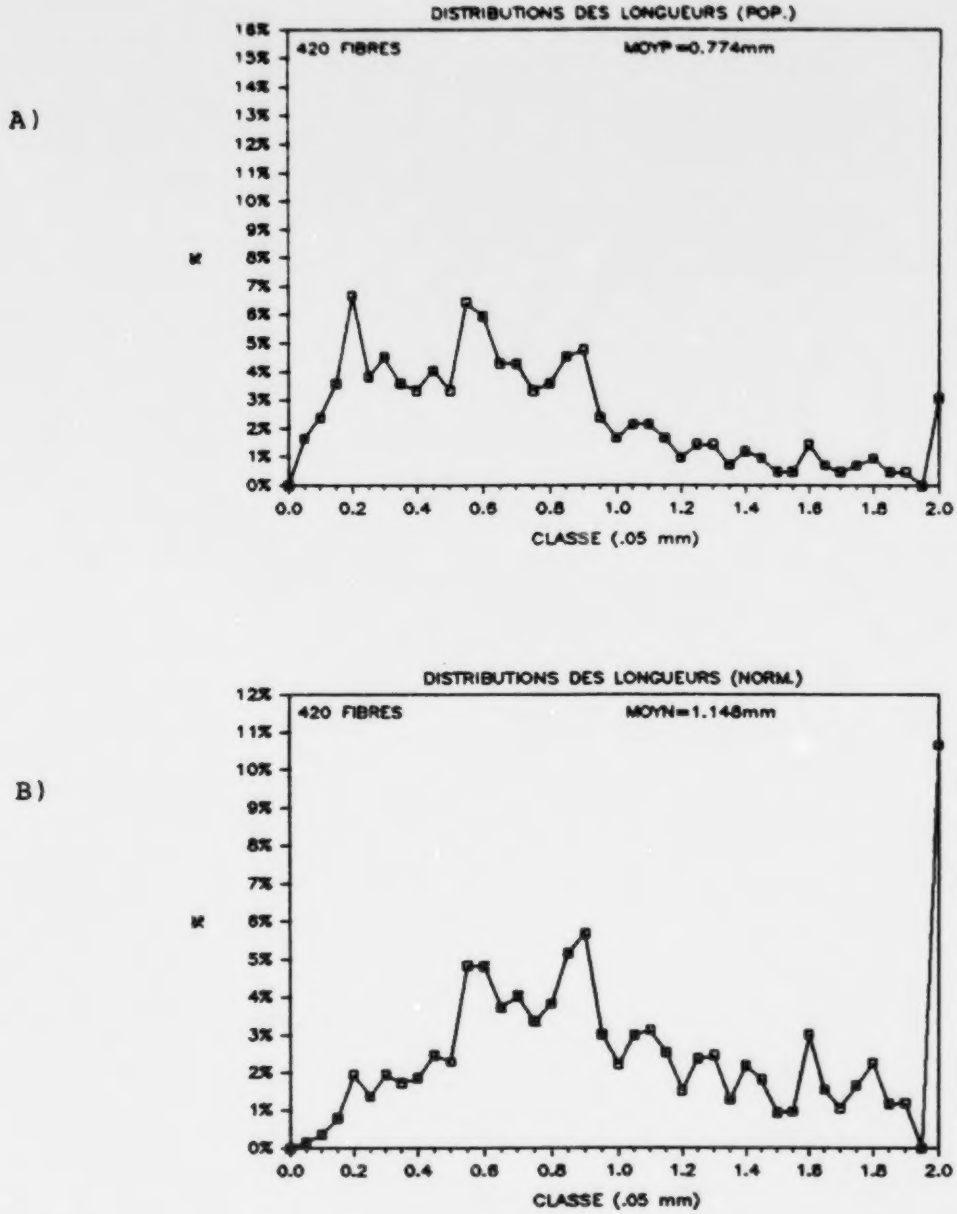


FIG. VIII.1c) Length distributions of L fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

CASSIAR WET-MILLED XL FIBERS (Bulk)

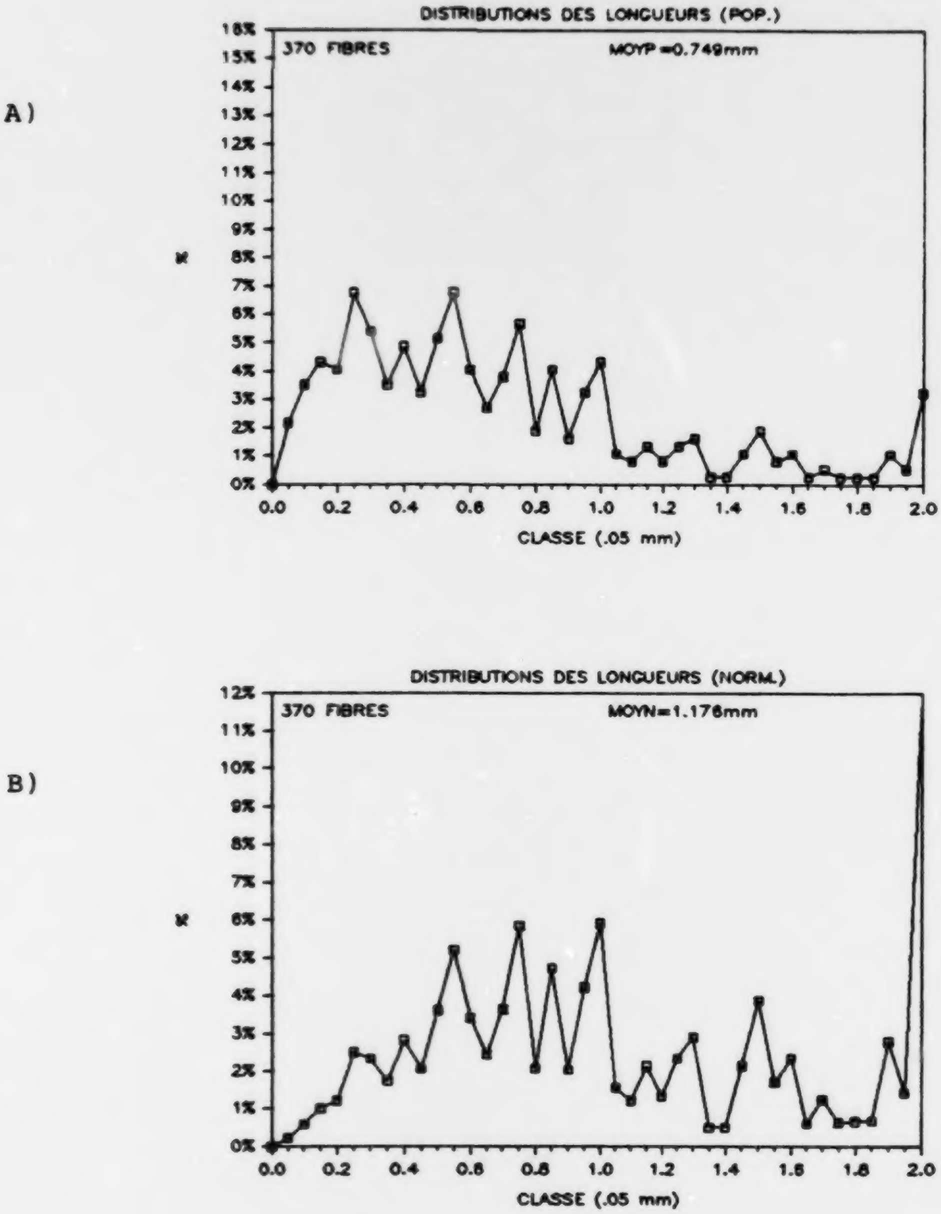


FIG. VIII.1d) Length distributions of XL fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

CASSIAR WET-MILLED FEED FIBERS (Bulk)

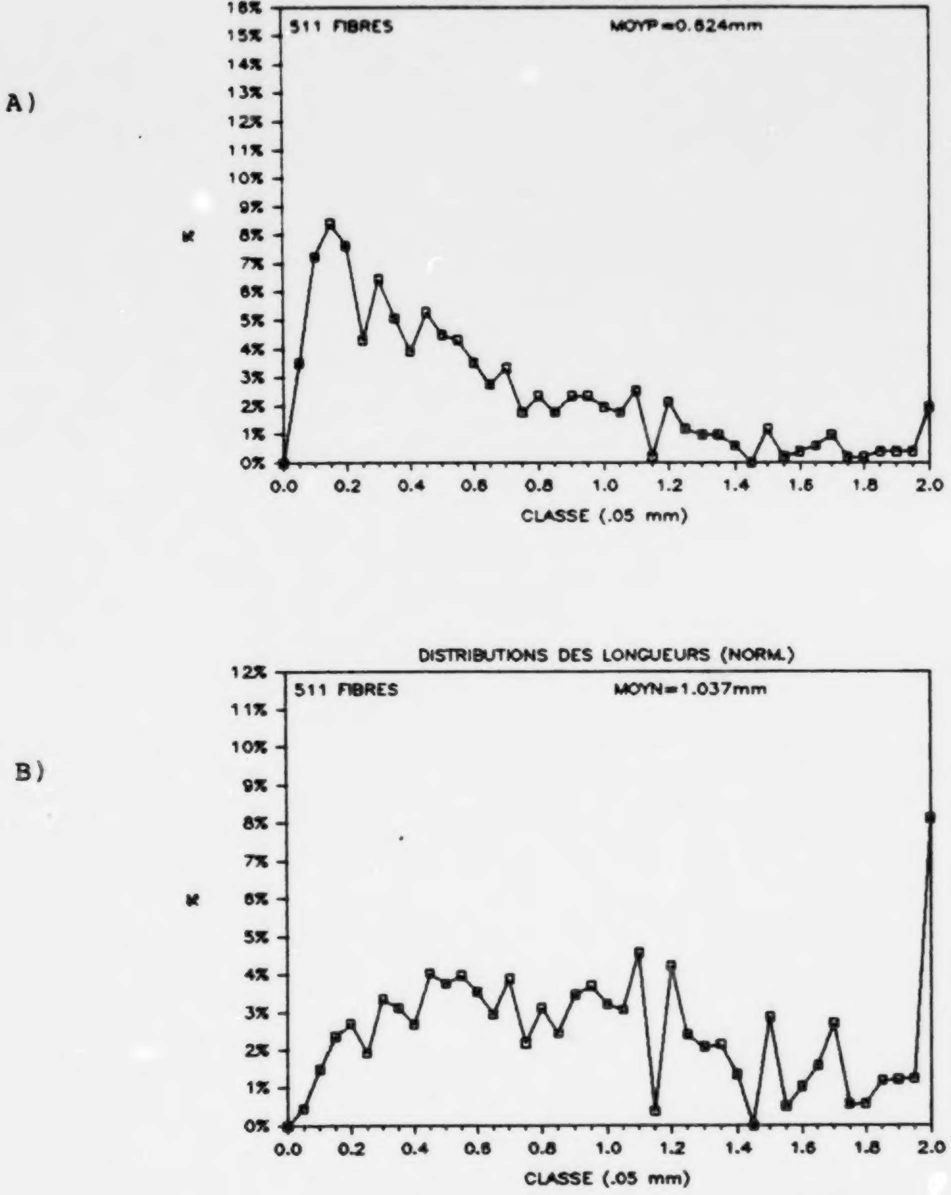


FIG. VIII.1e) Length distributions of FEED fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

WOODSREEF WET-MILLED 10-MESH FIBERS (Bulk)

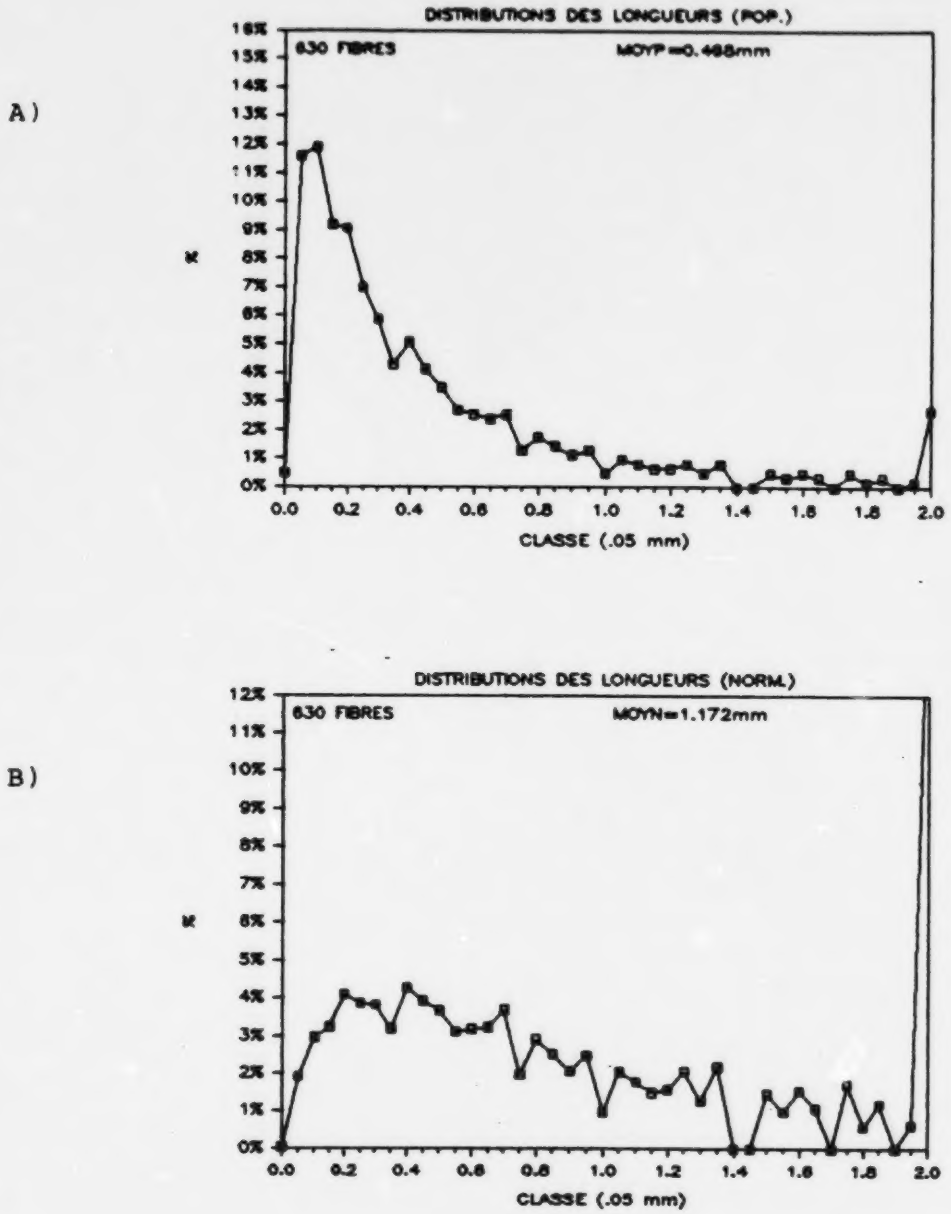


FIG. VIII.1f) Length distributions of Woodsreef 10-mesh fibers obtained through optical microscopy, by .05 mm classes

A) Population
B) Normalized

CASSIAR DRY-MILLED AZ FIBERS (Bulk)

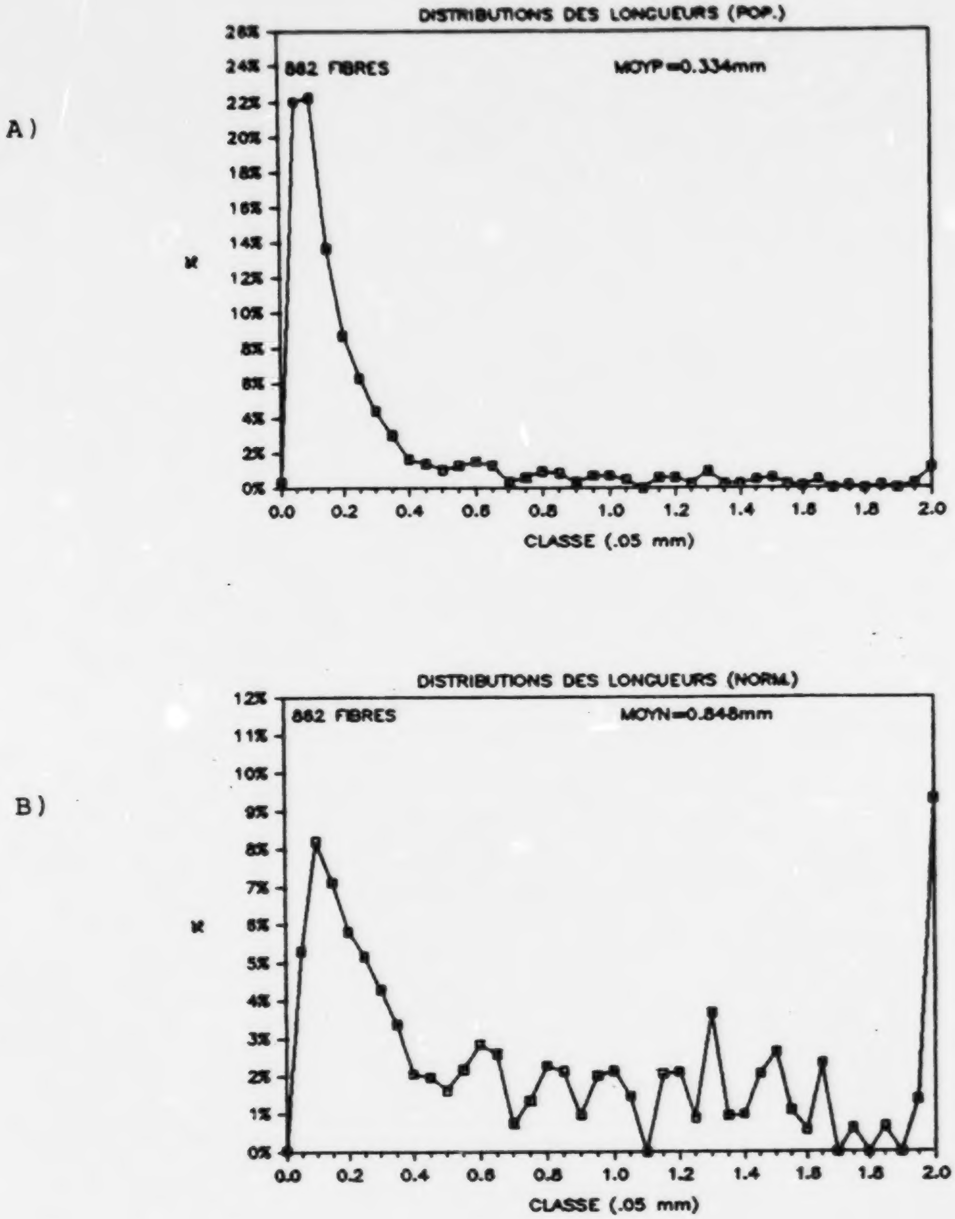


FIG. VIII.1g) Length distributions of AZ fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

CASSIAR DRY-MILLED AY FIBERS (Bulk)

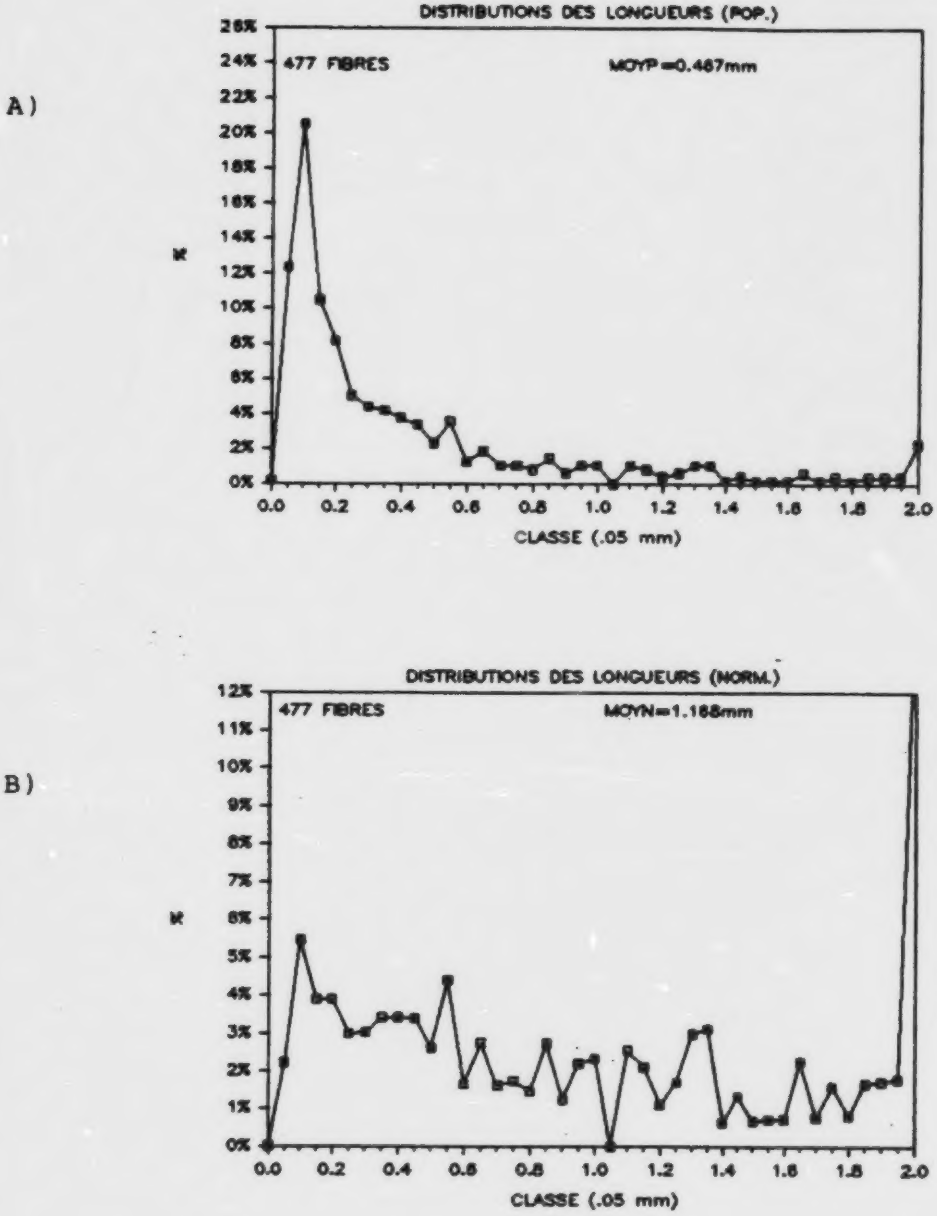
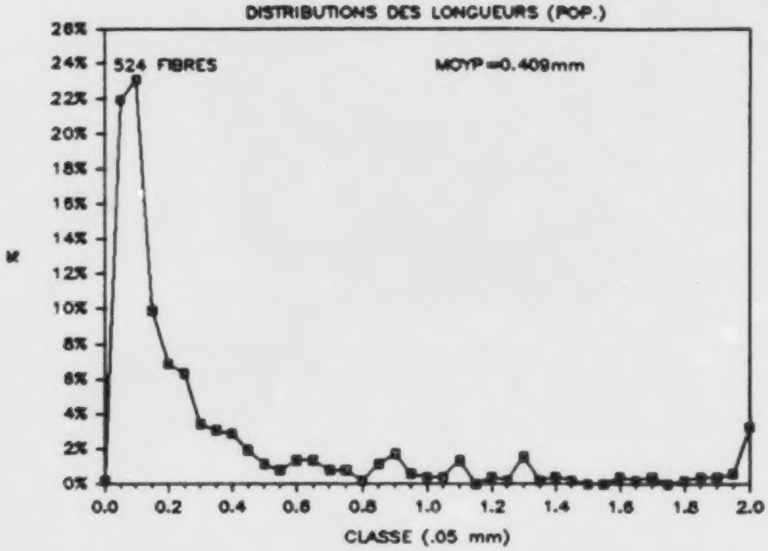


FIG. VIII.1h) Length distributions of AY fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

CASSIAR DRY-MILLED AX FIBERS (Bulk)

A)



B)

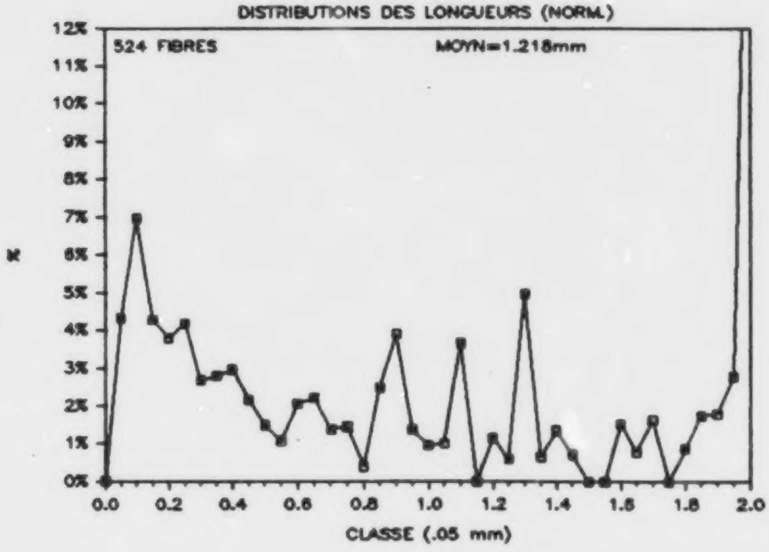


FIG. VIII.1i) Length distributions of AX fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

QUEBEC DRY-MILLED 4T FIBERS (Bulk)

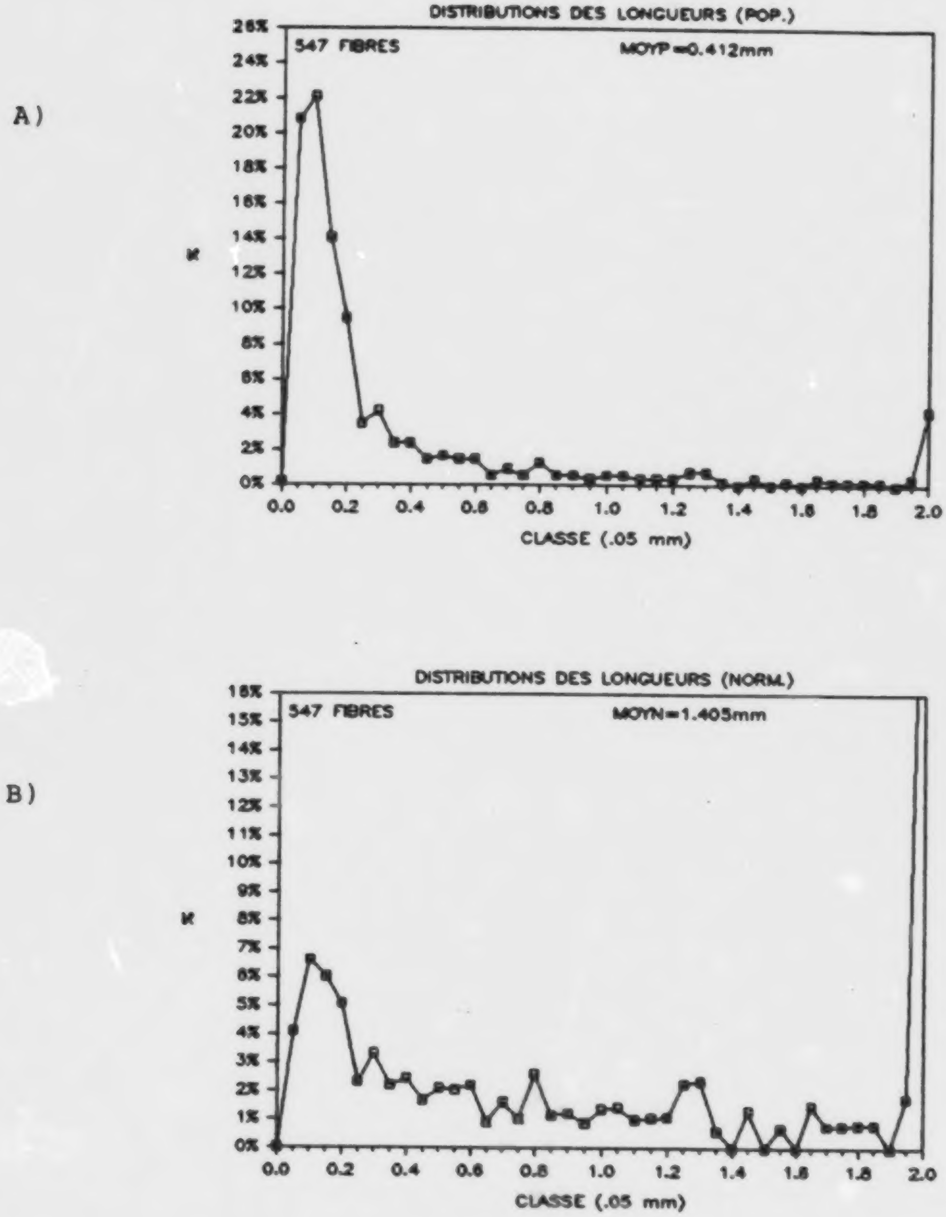


FIG. VIII.1J) Length distributions of 4T fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

The mean values of lengths and diameters are calculated using the following standard relations (where p represents either the lengths or the diameters).

mean values from population distributions

$$\bar{p} = \Sigma \ell / n$$

mean values from normalized distributions

$$\bar{p} = \Sigma \ell^2 / \Sigma \ell$$

The mean lengths ($\bar{\ell}$) and their standard deviations (σ_ℓ) computed for the various fiber types are collected in Table VIII.1.

TABLE VIII.1: Mean lengths ($\bar{\ell}$) and standard deviations (σ_ℓ) from OM length distributions of bulk fibers

Sample		No. of fibers	Population (mm)		Normalized (mm)
			$\bar{\ell}$	σ_ℓ	$\bar{\ell}$
Wet-milled	Cassiar S	461	.346	(.257)	.537
"	Cassiar M	532	.629	(.482)	.983
"	Cassiar L	420	.774	(.552)	1.148
"	Cassiar XL	370	.749	(.578)	1.176
"	Cassiar FEED	511	.624	(.511)	1.037
"	Woodsreef 10-mesh	630	.498	(.597)	1.172
Dry-milled	Cassiar AZ	862	.334	(.530)	.848
"	Cassiar AY	477	.467	(.584)	1.168
"	Cassiar AX	524	.409	(.587)	1.218
"	Quebec 4T	547	.412	(.658)	1.405

The population distribution data illustrate the following points:

- The mean lengths of all fiber types examined here fall in a relatively narrow range between 0.35 and 0.80 mm.

- The fiber length homogeneity as defined by the standard deviation σ_ℓ or $\sigma_\ell/\overline{\ell}$ is greater for the Cassiar wet-milled fibers than for all other types of fibers examined, including the wet-milled Woodsreef 10-mesh fibers.

- Among the Cassiar wet-milled fibers, the S fibers exhibit the greatest length homogeneity; the length homogeneity of the M, L and XL fibers is intermediate between the S and FEED products.

- The mean length $\overline{\ell}$ of the shortest and longest wet-classified fibers S and XL differ only by a factor of two, i.e., 0.35 and 0.75 μm.

b) Distributions of fiber diameters

Distributions of fiber diameters obtained through optical microscopy for the various fibers examined are illustrated in Fig. VIII.2a-j. The mean diameter values ($\overline{d}$) and standard deviations (σ_d) determined from these distributions are given in Table VIII.2.

CASSIAR WET-MILLED S FIBERS (Bulk)

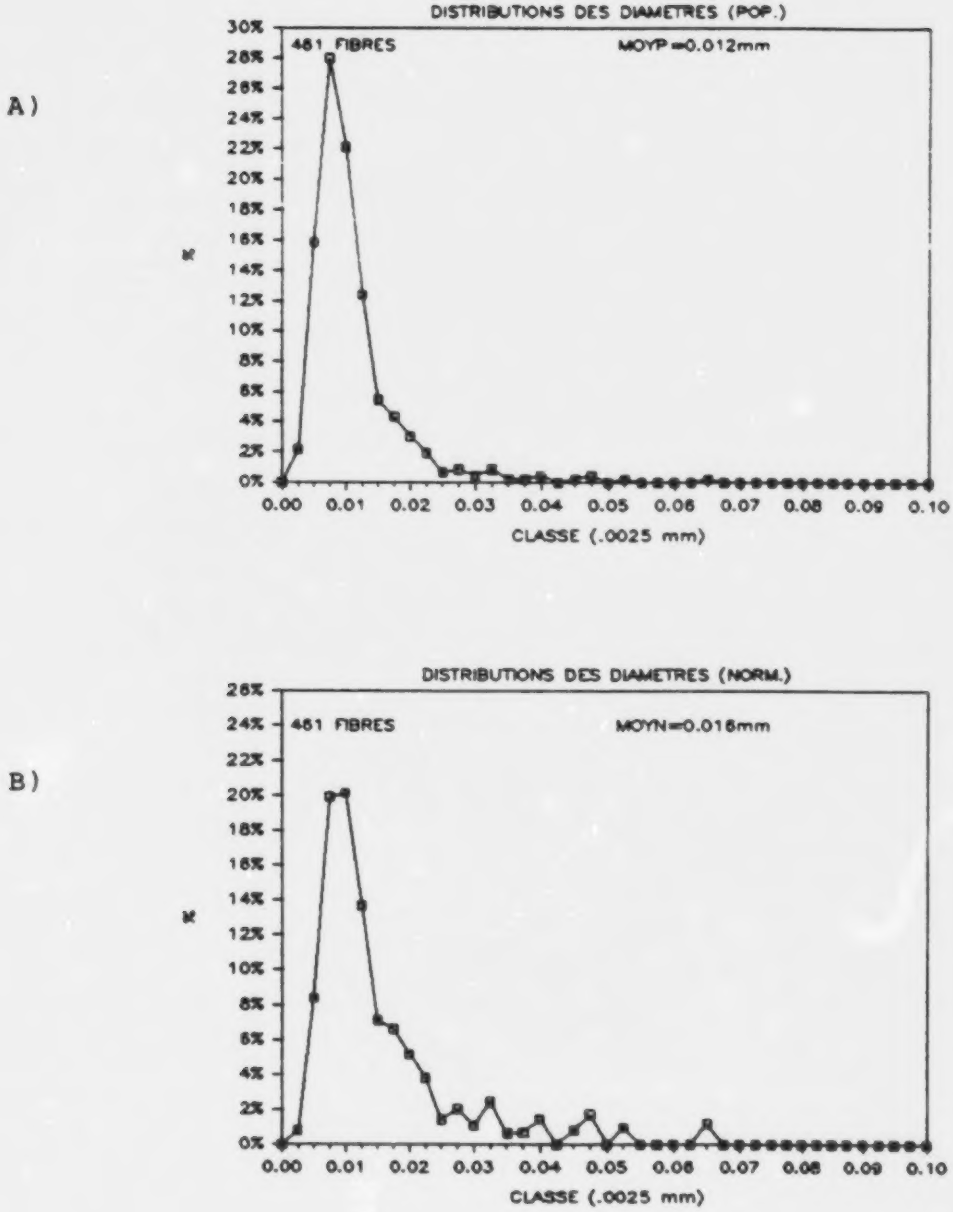


FIG. VIII.2a) Diameter distributions of S fibers obtained through optical microscopy, by .0025 mm classes.

A) Population
B) Normalized

(Class labels on X-coordinates give the lower limit of each class.)

CASSIAR WET-MILLED M FIBERS (Bulk)

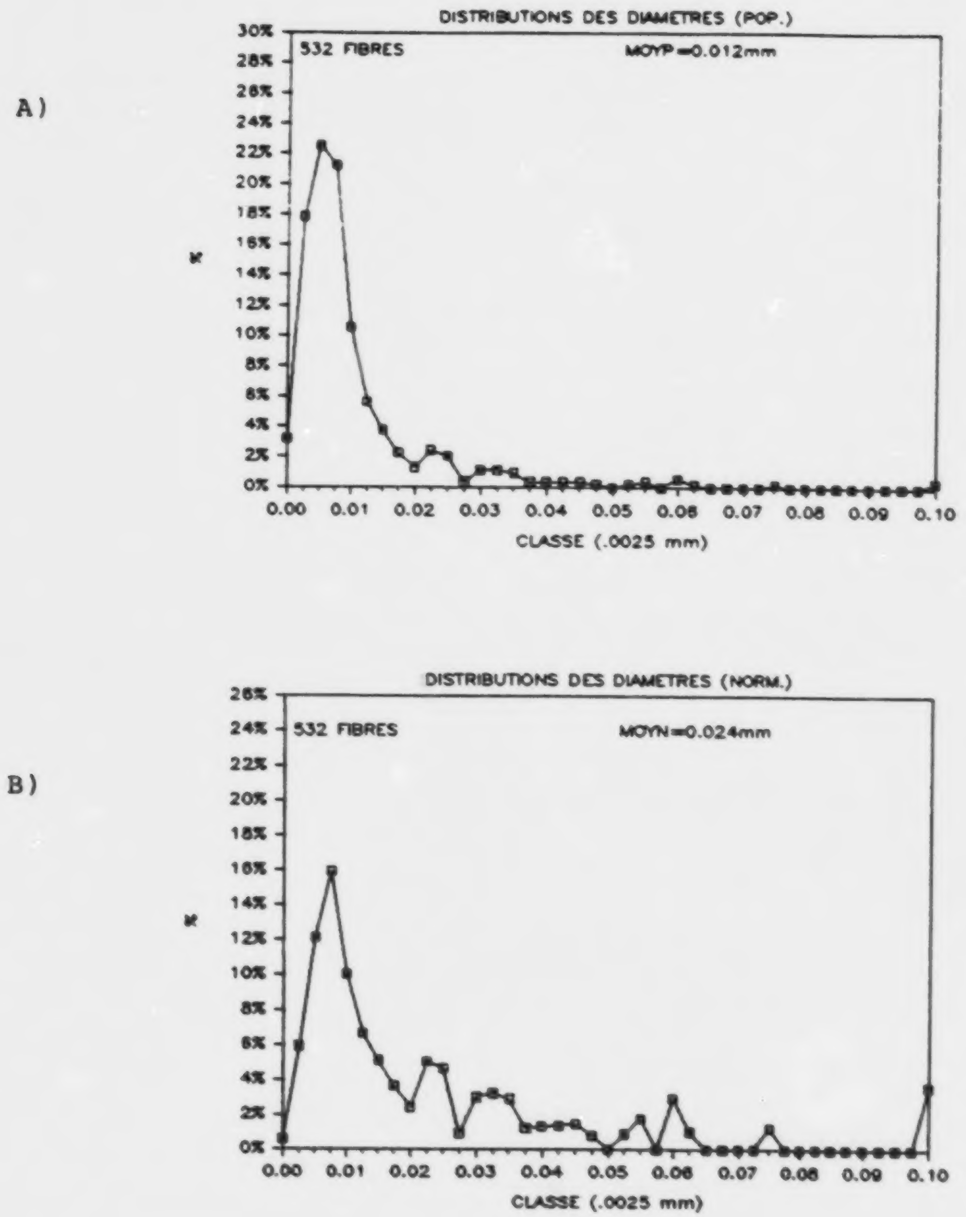


FIG. VIII.2b) Diameter distributions of M fibers obtained through optical microscopy, by .0025 mm classes.

A) Population
B) Normalized

CASSIAR WET-MILLED L FIBERS (Bulk)

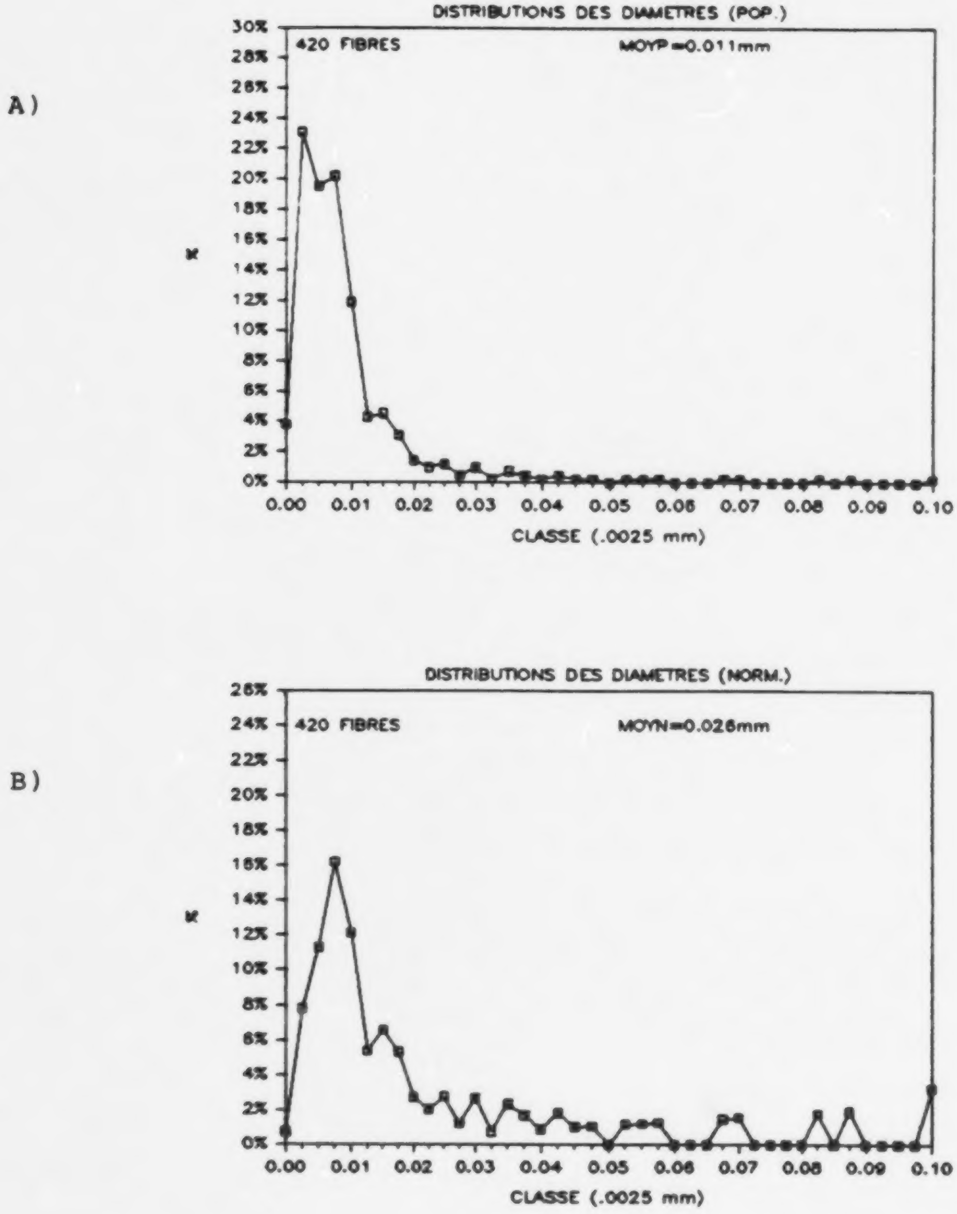


FIG. VIII.2c) Diameter distributions of L fibers obtained through optical microscopy, by .0025 mm classes.

A) Population
B) Normalized

CASSIAR WET-MILLED XL FIBERS (Bulk)

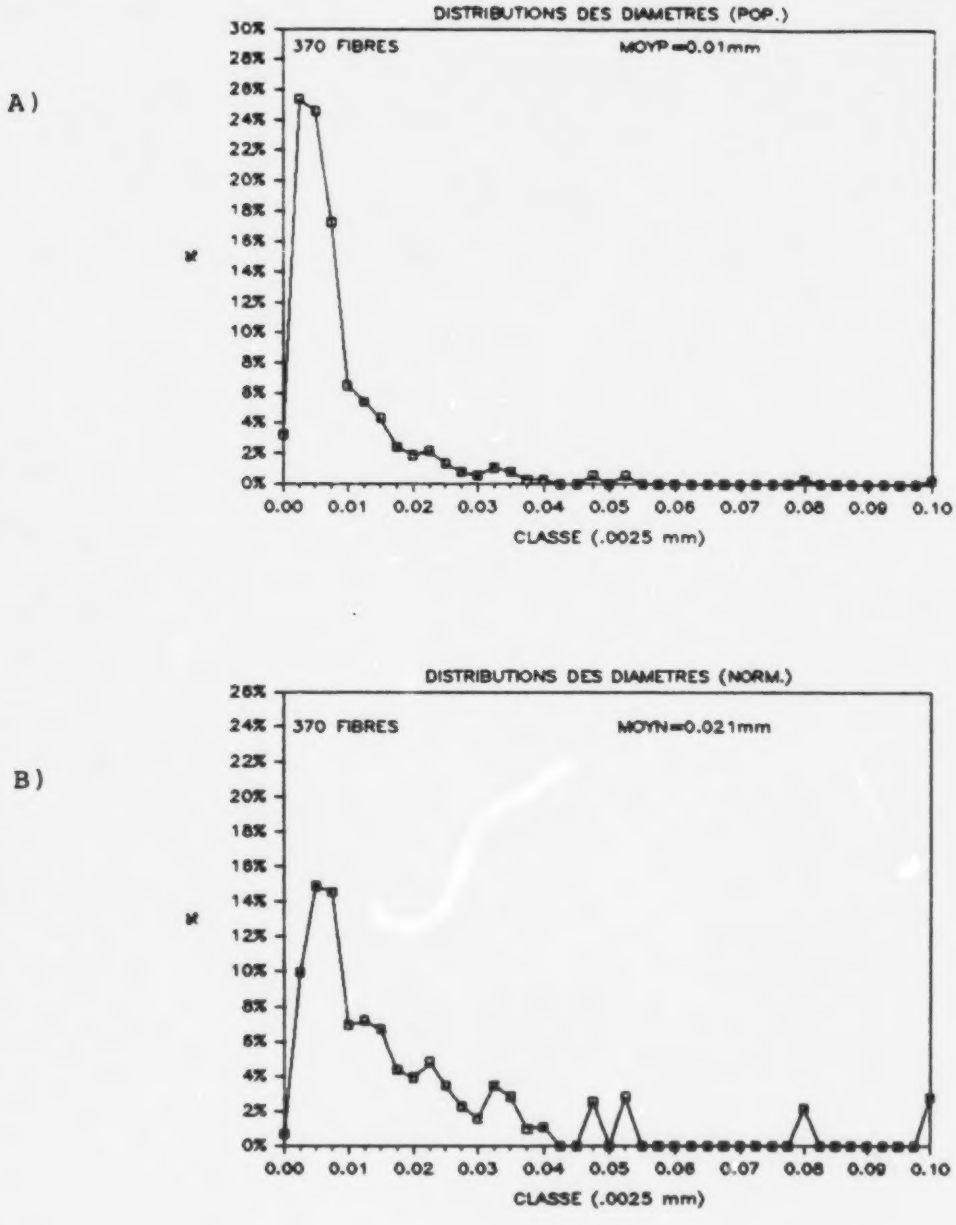


FIG. VIII.2d) Diameter distributions of XL fibers obtained through optical microscopy, by .0025 mm classes.

A) Population
B) Normalized

CASSIAR WET-MILLED FEED FIBERS (Bulk)

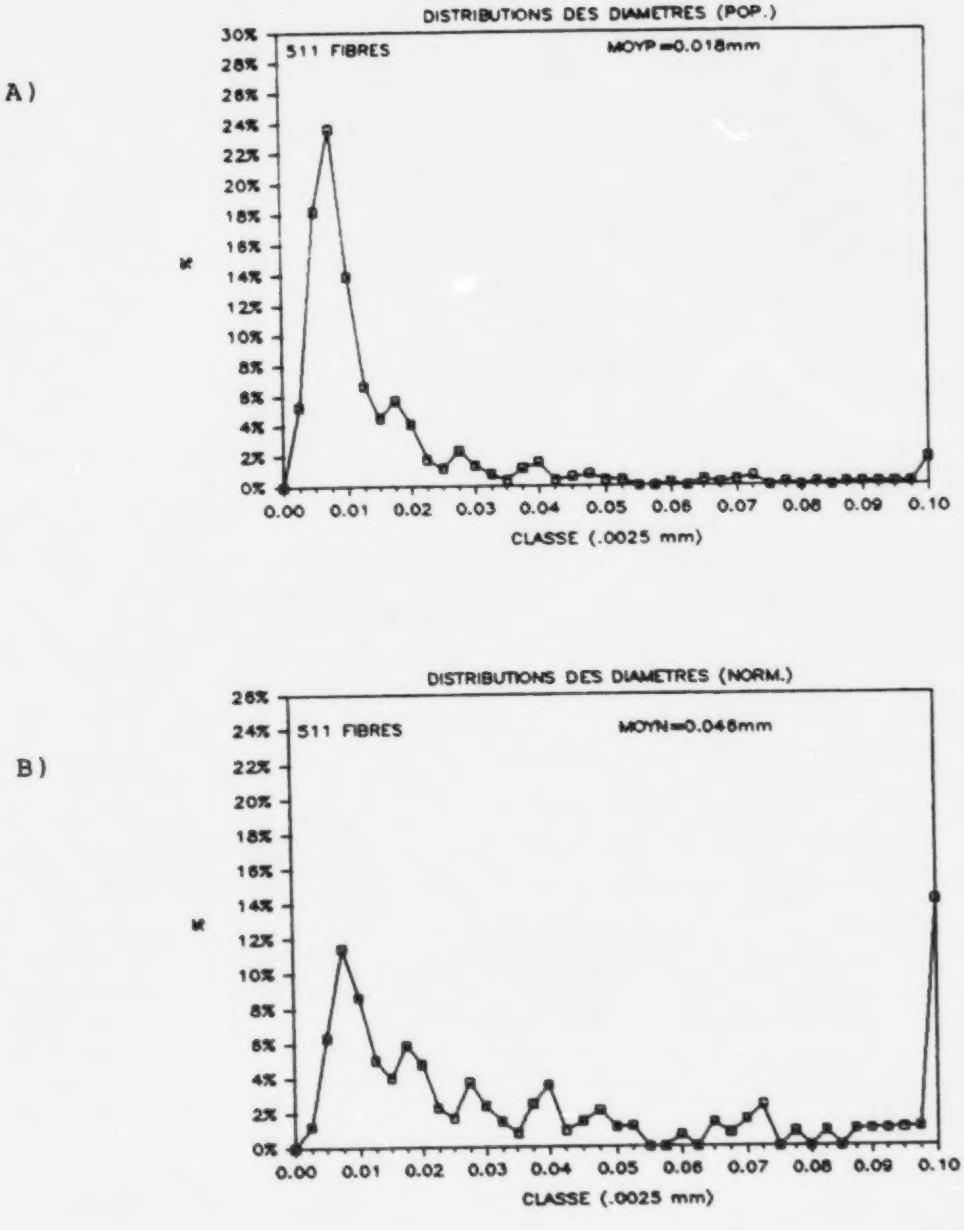


FIG. VIII.2e) Diameter distributions of FEED fibers obtained through optical microscopy, by .0025 mm classes.

A) Population
B) Normalized

WOODSREEF WET-MILLED 10-MESH FIBERS (Bulk)

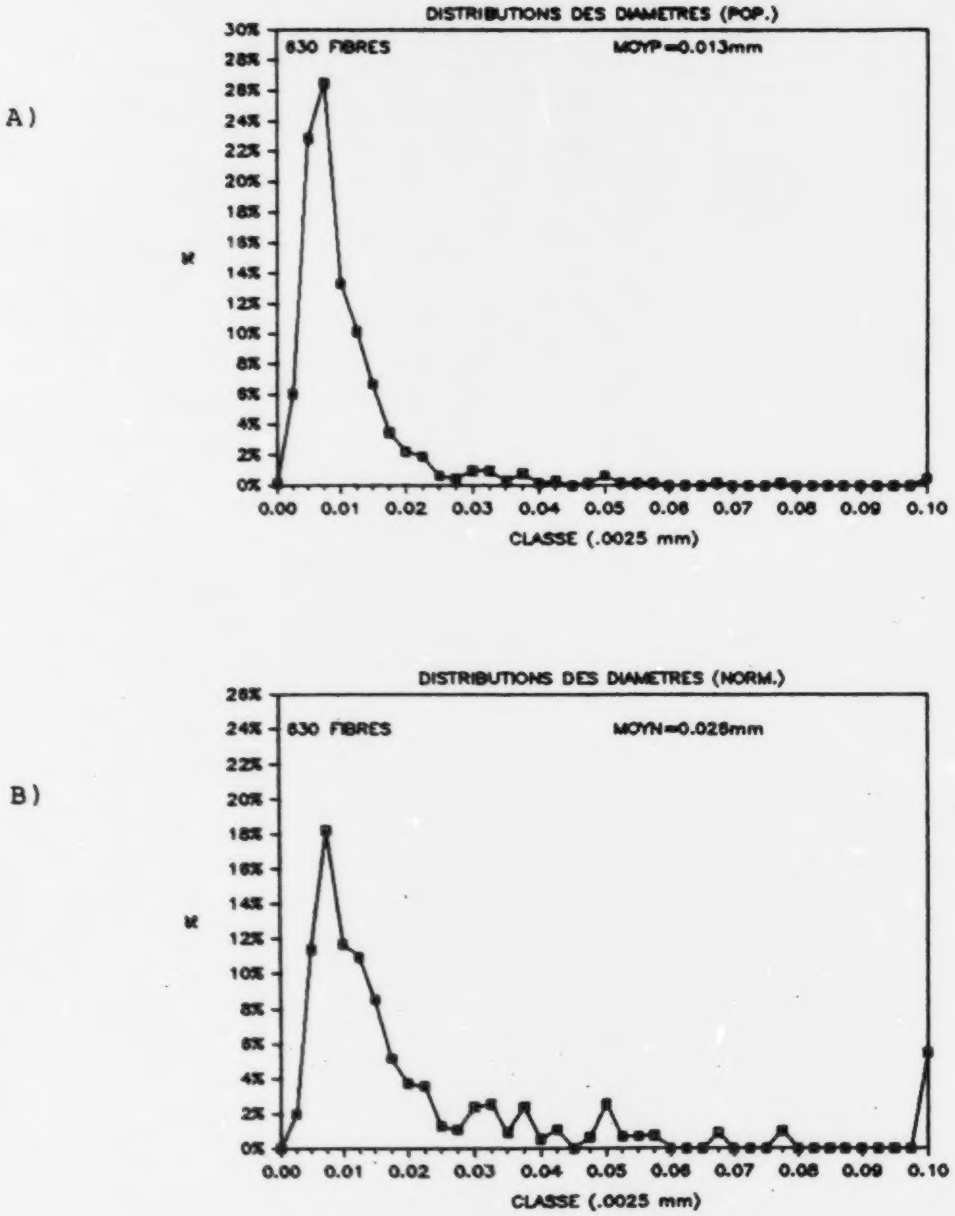


FIG. VIII.2f) Diameter distributions of Woodsreef 10-mesh fibers obtained through optical microscopy, by .0025 mm classes.

A) Population
B) Normalized

CASSIAR DRY-MILLED AZ FIBERS (Bulk)

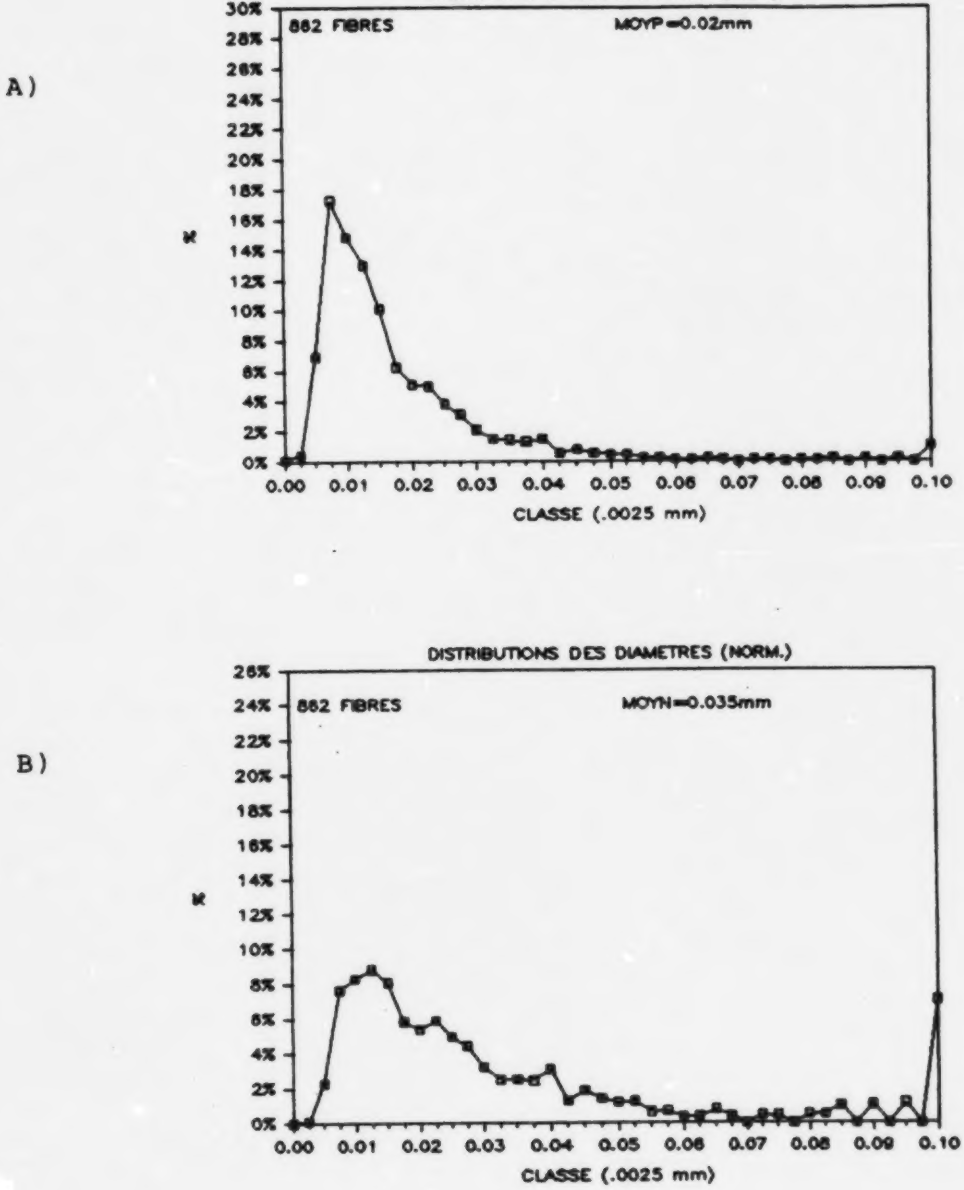


FIG. VIII.2g) Diameter distributions of AZ fibers obtained through optical microscopy, by .0025 mm classes.

A) Population
B) Normalized

CASSIAR DRY-MILLED AY FIBERS (Bulk)

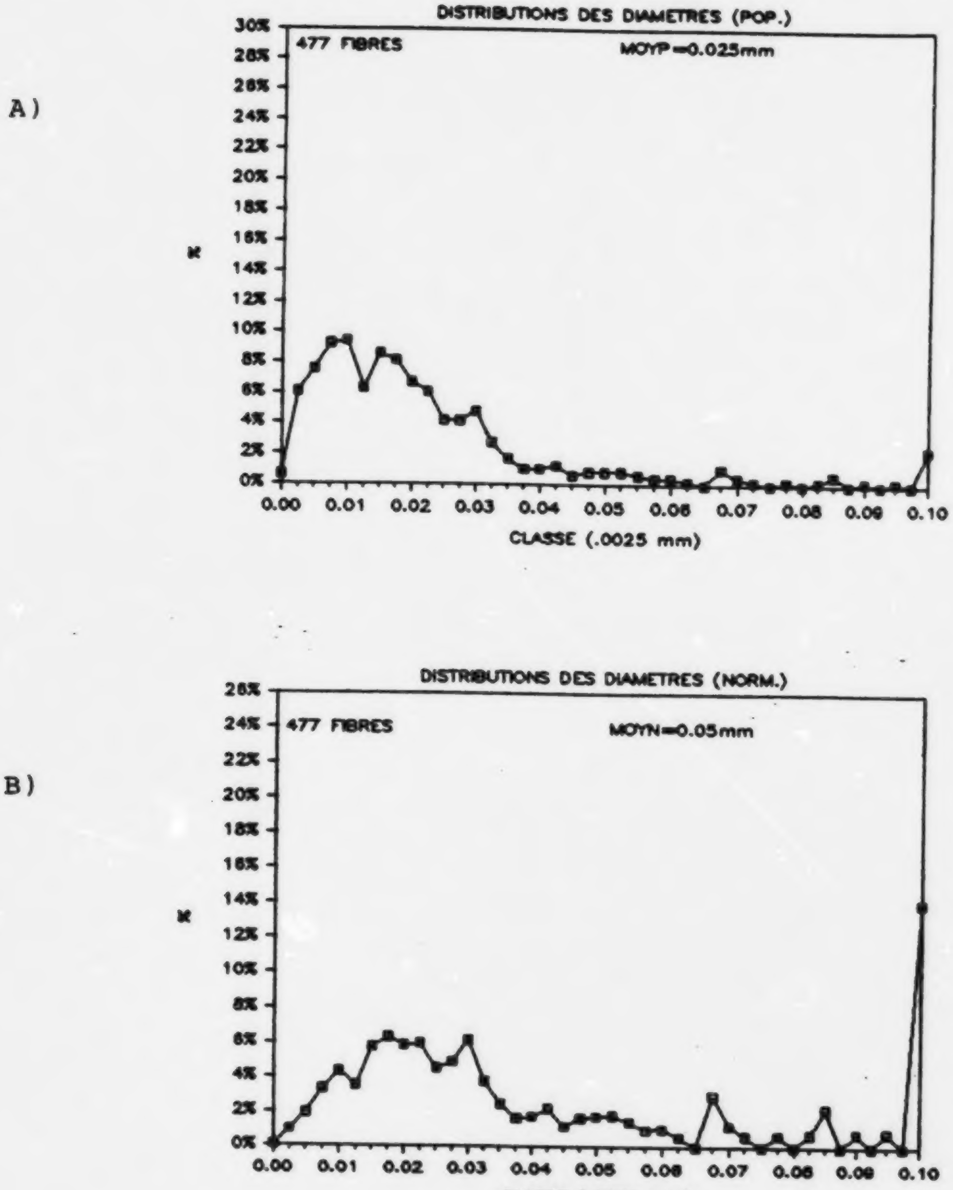


FIG. VIII.2h) Diameter distributions of AY fibers obtained through optical microscopy, by .0025 mm classes.

A) Population
B) Normalized

CASSIAR DRY-MILLED AX FIBERS (Bulk)

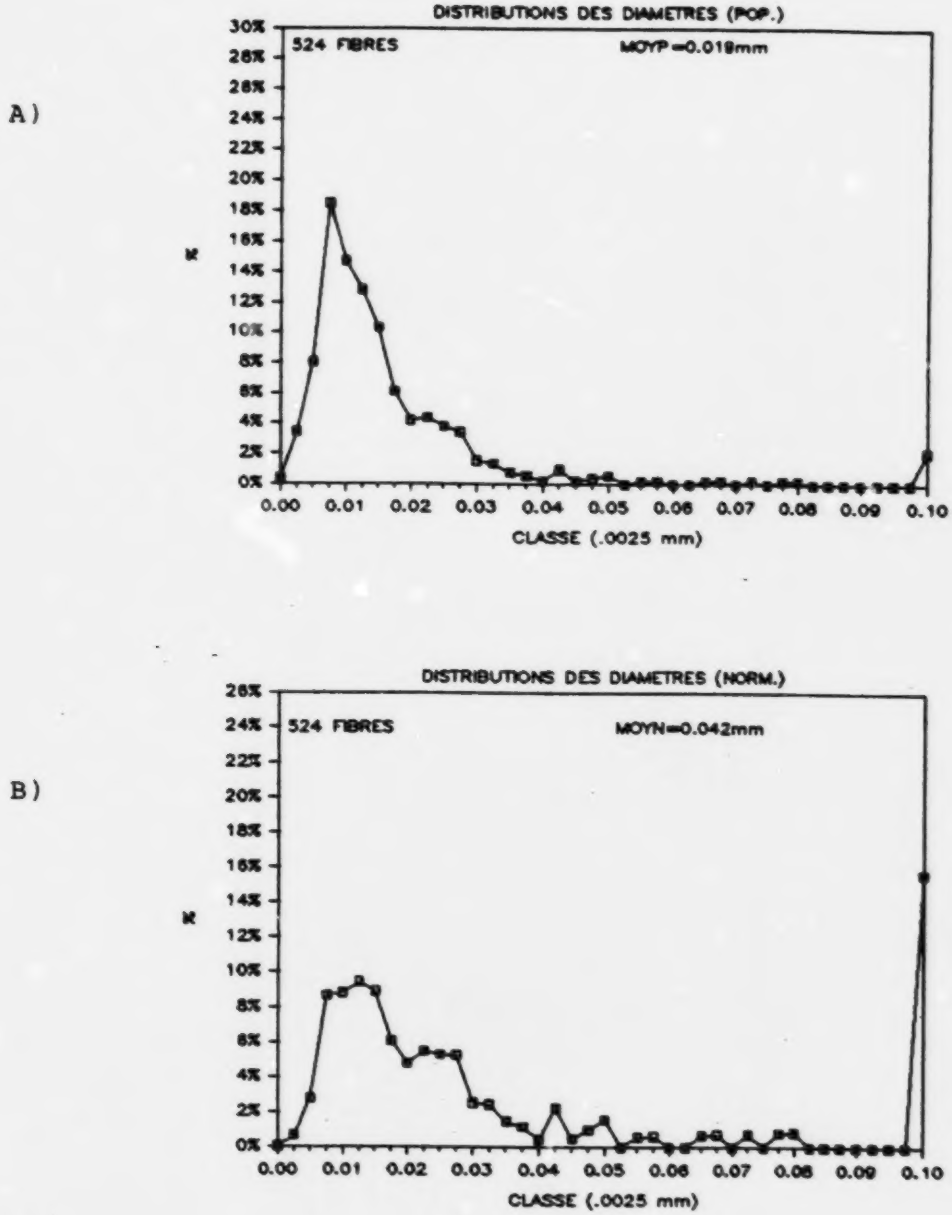


FIG. VIII.2i) Diameter distributions of AX fibers obtained through optical microscopy, by .0025 mm classes.

A) Population
B) Normalized

QUEBEC DRY-MILLED 4T FIBERS (Bulk)

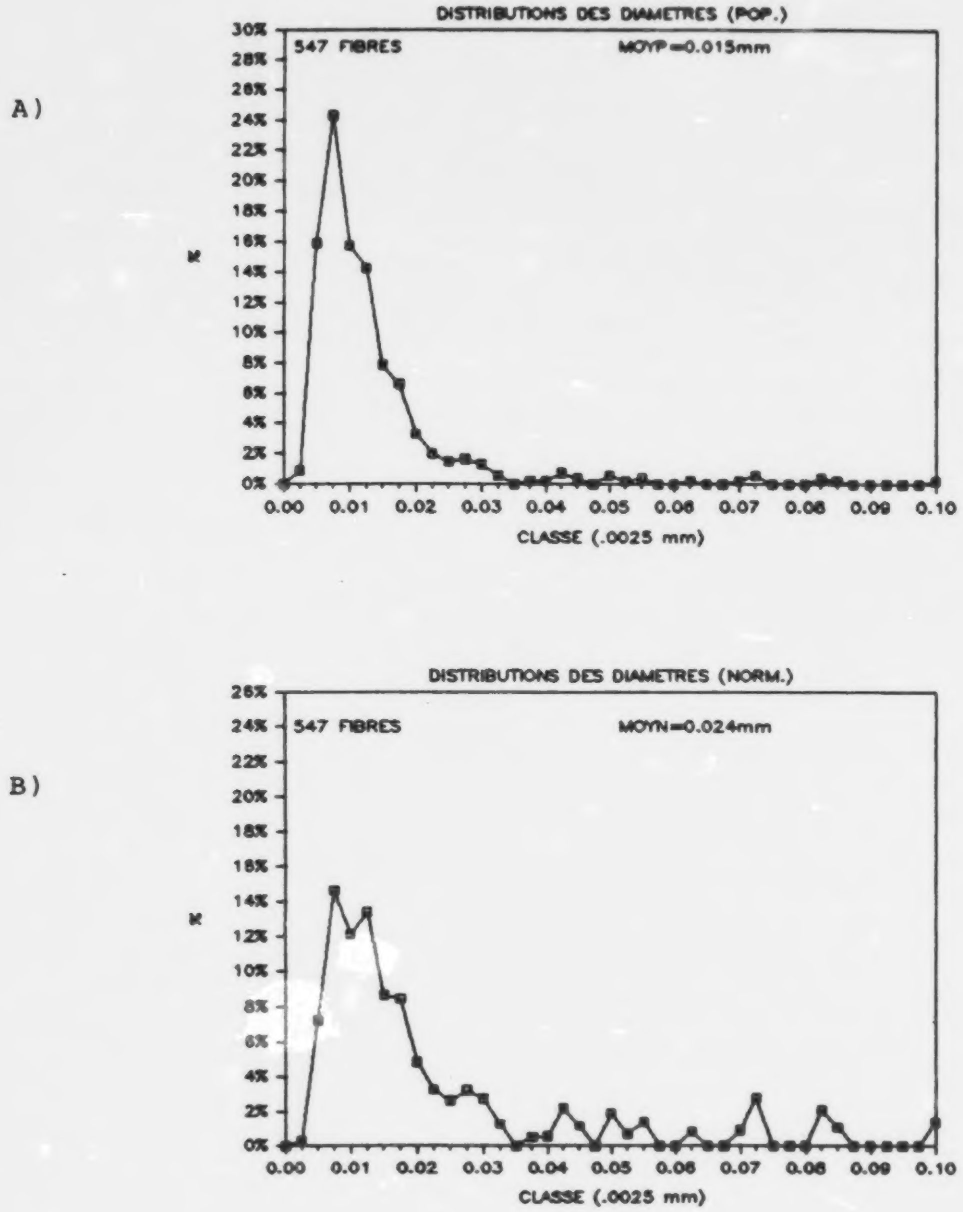


FIG. VIII.2j) Diameter distributions of 4T fibers obtained through optical microscopy, by .0025 mm classes.

A) Population
B) Normalized

TABLE VIII.2: Mean diameters (d) and standard deviations (σ_d) from OM diameter distributions of bulk fibers

Sample	No. of fibers	Population (mm)		Normalized (mm)
		$\bar{d}$	σ_d	$\bar{d}$
Wet-milled Cassiar S	461	.012	(.007)	.016
" Cassiar M	532	.012	(.012)	.024
" Cassiar L	420	.011	(.013)	.026
" Cassiar XL	370	.010	(.010)	.021
" Cassiar FEED	511	.018	(.023)	.046
" Woodsreef 10-mesh	630	.013	(.013)	.026
Dry-milled Cassiar AZ	862	.020	(.025)	.035
" Cassiar AY	477	.025	(.026)	.050
" Cassiar AX	524	.019	(.021)	.042
" Quebec 4T	527	.015	(.012)	.024

An examination of the population distribution data points to the following:

- The mean diameters of all fiber types examined here fall in the range 0.10-0.25 mm.

- The average diameters of the wet-milled fibers (Cassiar or Woodsreef) are systematically lower than those of dry-milled products.

- The mean diameters of the Cassiar wet-milled classified fibers (S, M, L, XL) fall within a very narrow range: 11 ± 1 μm.

- The mean diameter of the FEED fibers is substantially larger ($\sim$ 50%) than the mean values of the wet-classified fibers; this most certainly reflects the fact that the classification technology used induces a significant degree of fiber opening.

- The homogeneity of fiber diameters as reflected by σ_d is greater for the wet-milled fibers than for the dry-milled fibers ($\sigma_d/\overline{d}$ values are however comparable in all fiber types).

- Judging by the values of σ_d or $\sigma_d/\overline{d}$ for the Cassiar wet-milled fibers, the diameter homogeneity is highest for the S fibers and lowest for the FEED fibers; the diameter homogeneity of M, L and XL fibers are comparable and intermediate between those of S and FEED fibers.

VIII.2 - Results for Pallmann opened fibers

In this section, we report the distributions of fiber lengths and diameters for the various fiber products following opening on a Pallmann mill at 6500 rpm. As for the bulk fiber products, the distributions were obtained from measurements on 47X magnification optical microscopy photographs.

a) Fiber length distributions

The fiber length distributions (population and normalized) obtained for Pallmann opened fibers are reproduced in Fig. VIII.3a-j; the mean length values (ℓ) and standard deviations (σ_d) are collected in VIII.3.

TABLE VIII.3: Mean length ($\overline{\ell}$) and standard deviations (σ_ℓ) from OM length distributions of Pallmann opened fibers

Sample		No. of fibers	Population (mm) $\overline{\ell}$	Population (mm) σ_ℓ	Normalized (mm) $\overline{\ell}$
Wet-milled	Cassiar S	386	.380	(.318)	.646
"	Cassiar M	631	.596	(.435)	.911
"	Cassiar L	493	.666	(.467)	.993
"	Cassiar XL	595	.727	(.532)	1.108
"	Cassiar FEED	462	.522	(.408)	.838
"	Woodsreef 10-Mesh	420	.443	(.408)	.932
Dry-milled	Cassiar AZ	789	.421	(.456)	.900
"	Cassiar AY	630	.486	(.626)	1.263
"	Cassiar AX	648	.557	(.770)	1.408
"	Quebec 4T	714	.443	(.640)	1.118

CASSIAR WET-MILLED S FIBERS (Open)

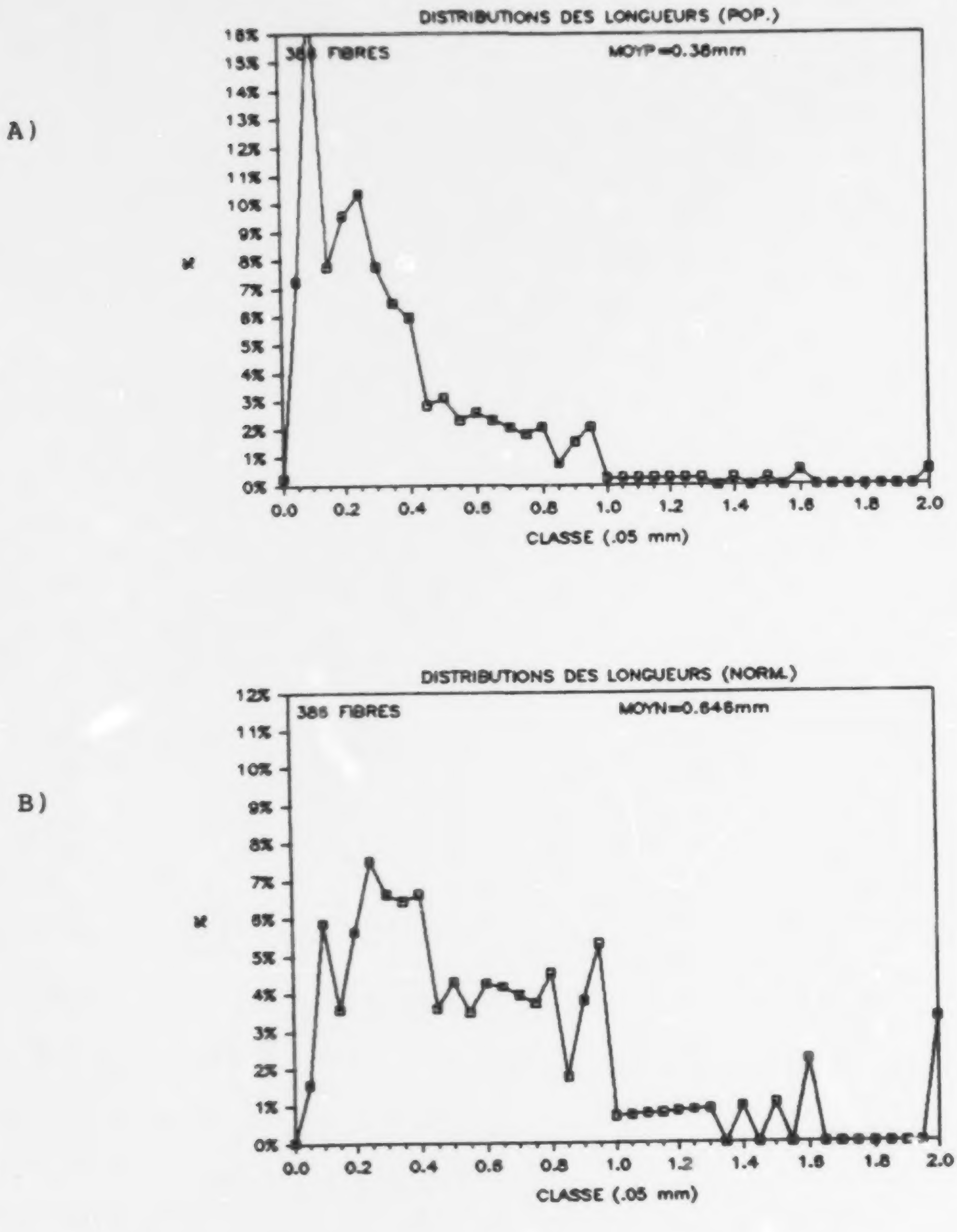


FIG. VIII.3a) Length distributions of S Pallmann opened fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

(Class labels on X-coordinates give the lower limit of each class.)

CASSIAR WET-MILLED M FIBERS (Open)

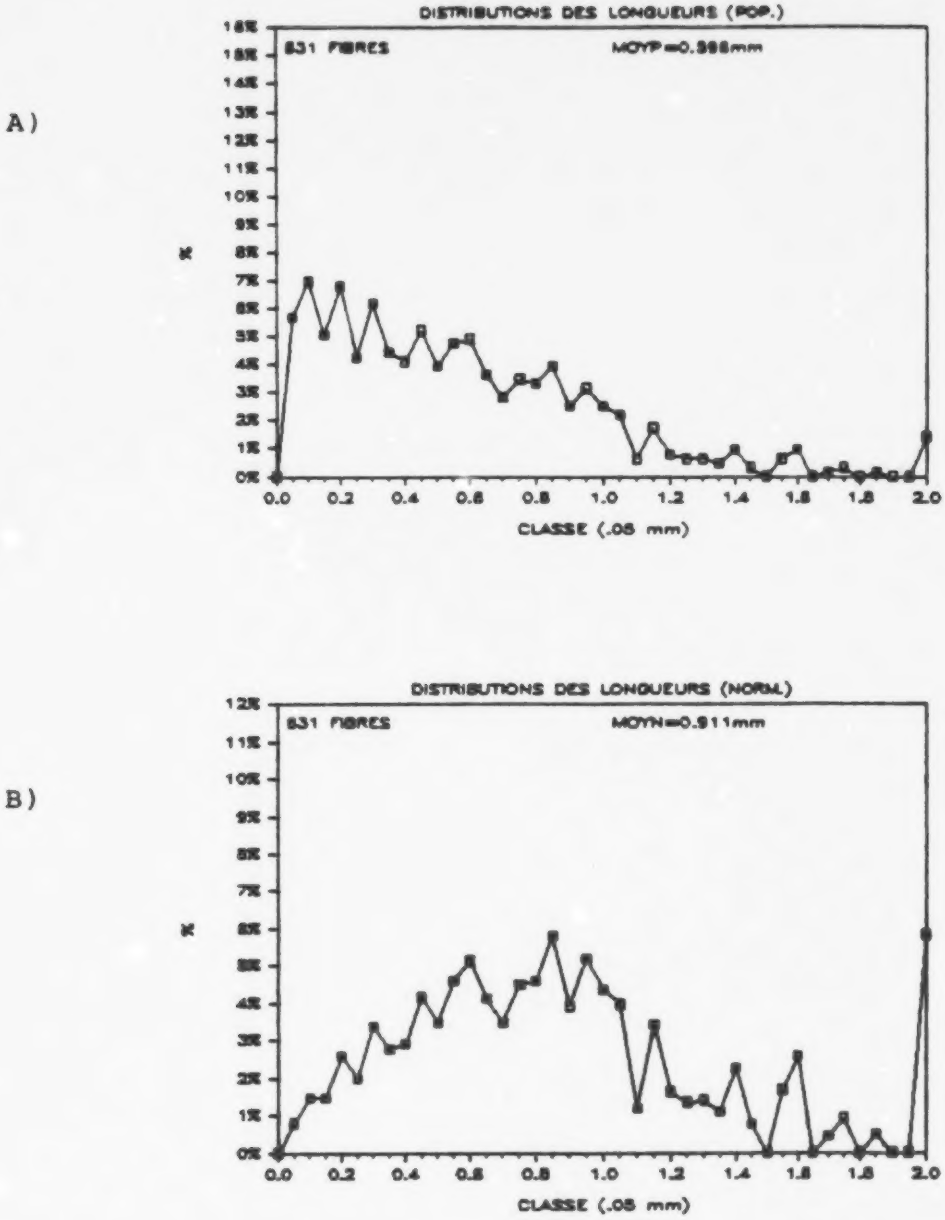


FIG. VIII.3b) Length distributions of M Pallmann opened fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

CASSIAR WET-MILLED L FIBERS (Open)

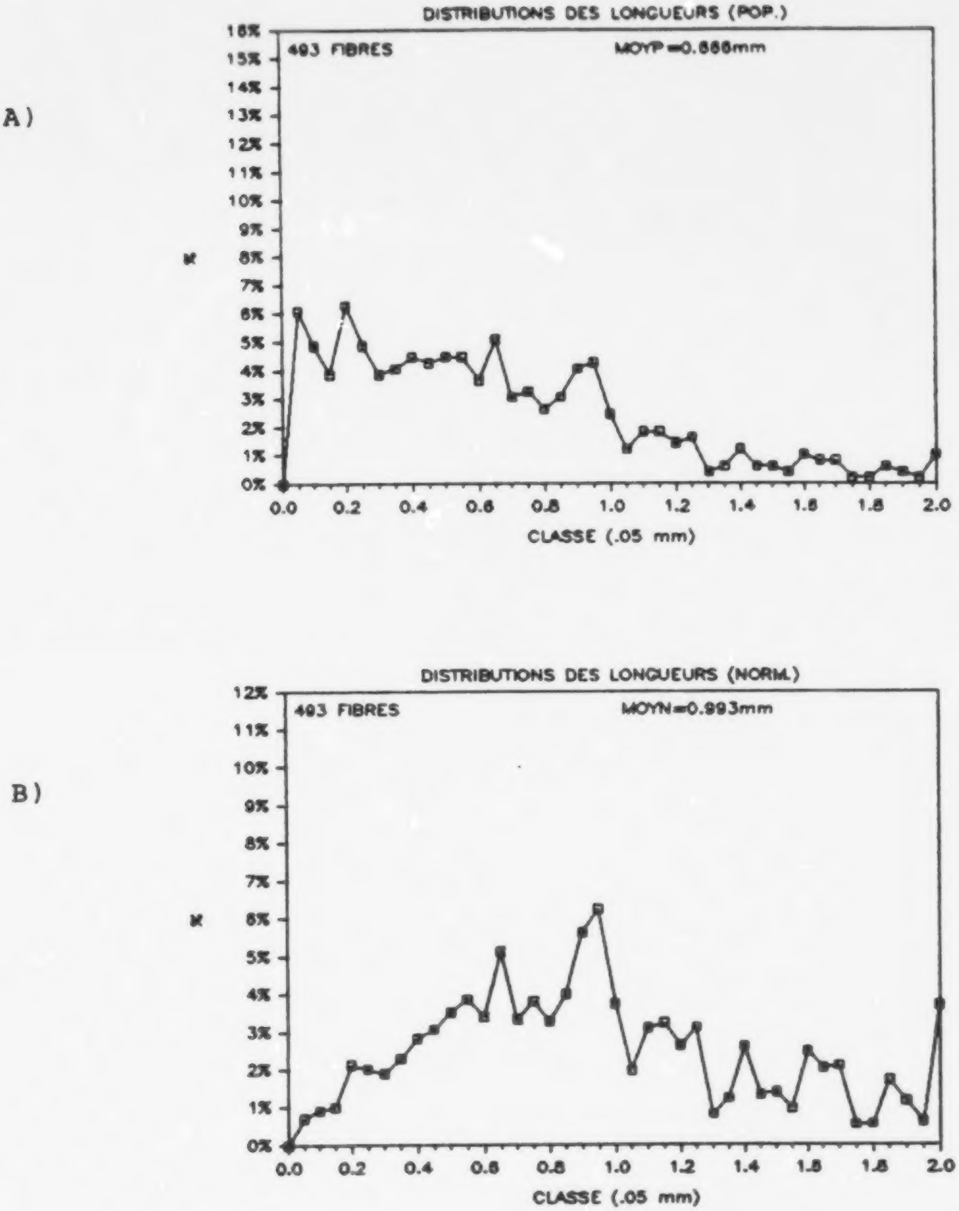


FIG. VIII.3c) Length distributions of L Pallmann opened fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

CASSIAR WET-MILLED XL FIBERS (Open)

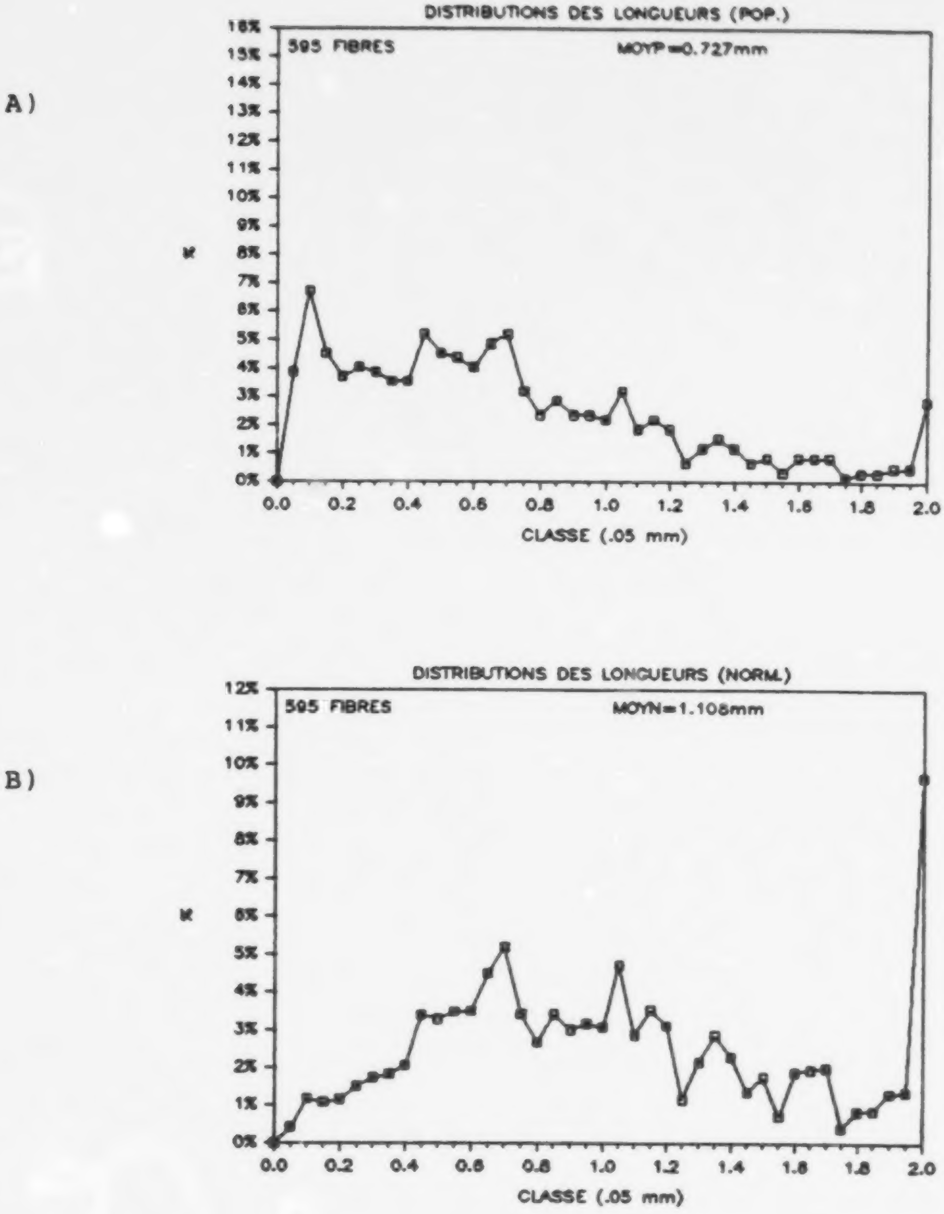


FIG. VIII.3d) Length distributions of XL Pallmann opened fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

CASSIAR WET-MILLED FEED FIBERS (Open)

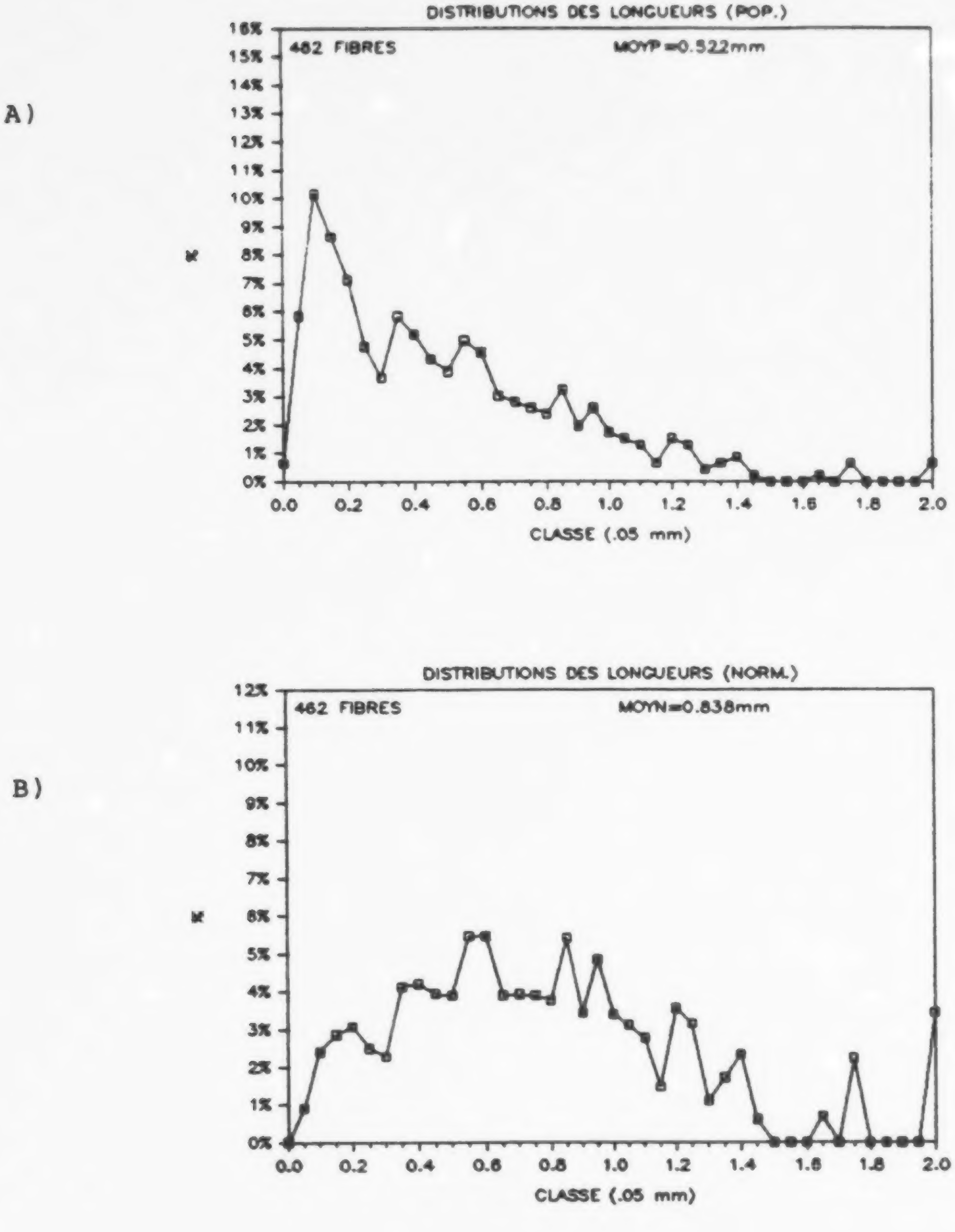


FIG. VIII.3e) Length distributions of FEED Pallmann opened fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

WOODSREEF WET-MILLED 10-MESH FIBERS (Open)

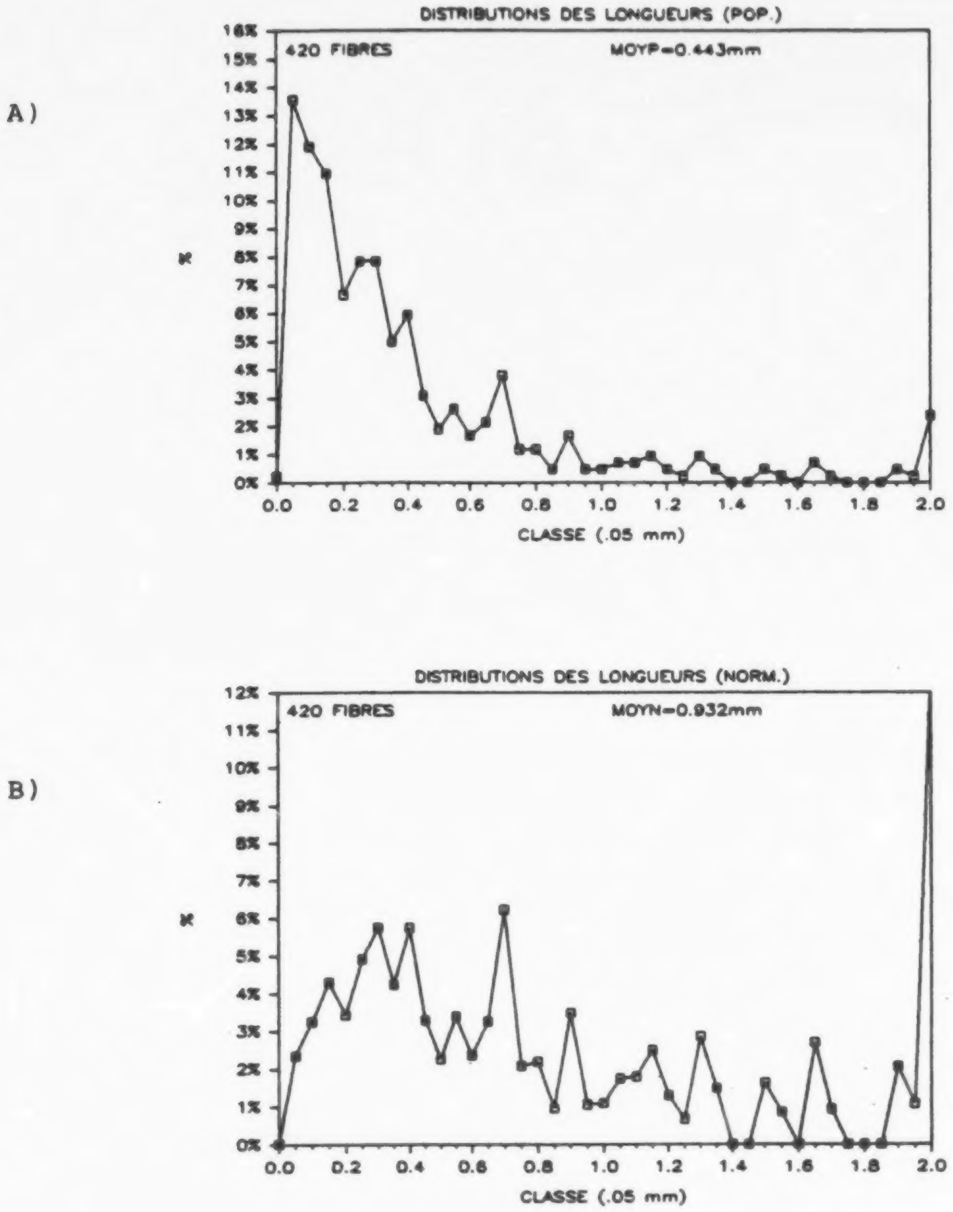


FIG. VIII.3f) Length distributions of Woodsreef 10-mesh Pallmann opened fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

CASSIAR DRY-MILLED AZ FIBERS (Open)

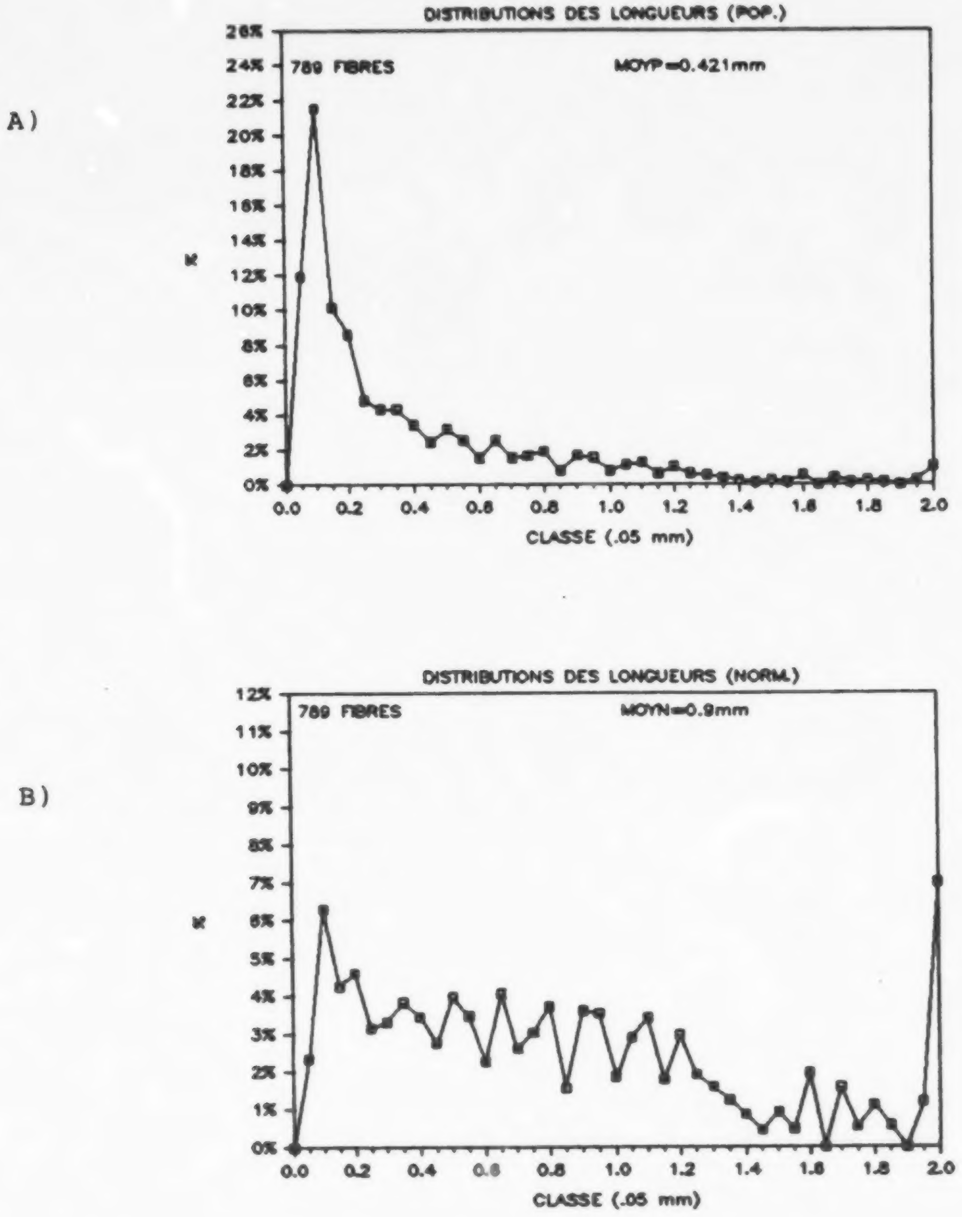
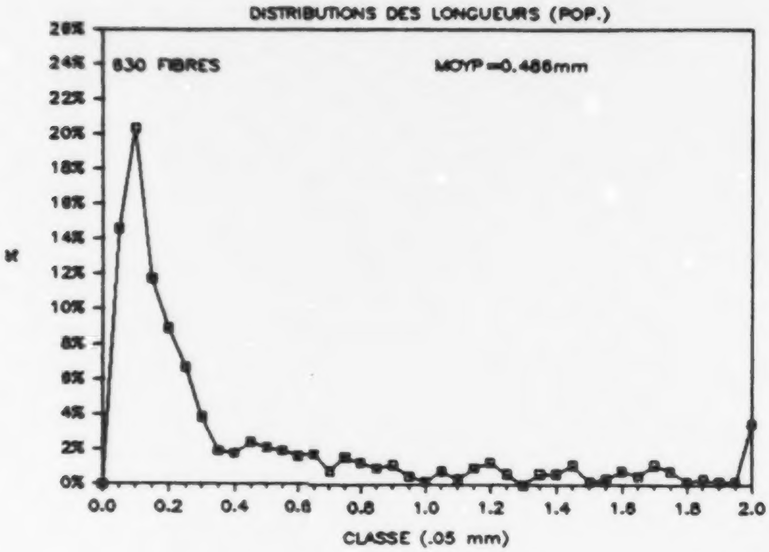


FIG. VIII.3g) Length distributions of AZ Pallmann opened fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

CASSIAR DRY-MILLED AY FIBERS (Open)

A)



B)

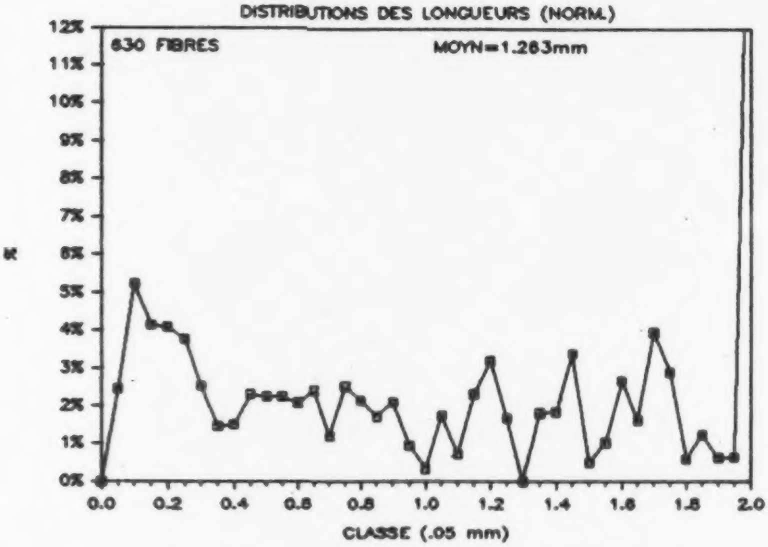


FIG. VIII.3h) Length distributions of AY Pallmann opened fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

CASSIAR DRY-MILLED AX FIBERS (Open)

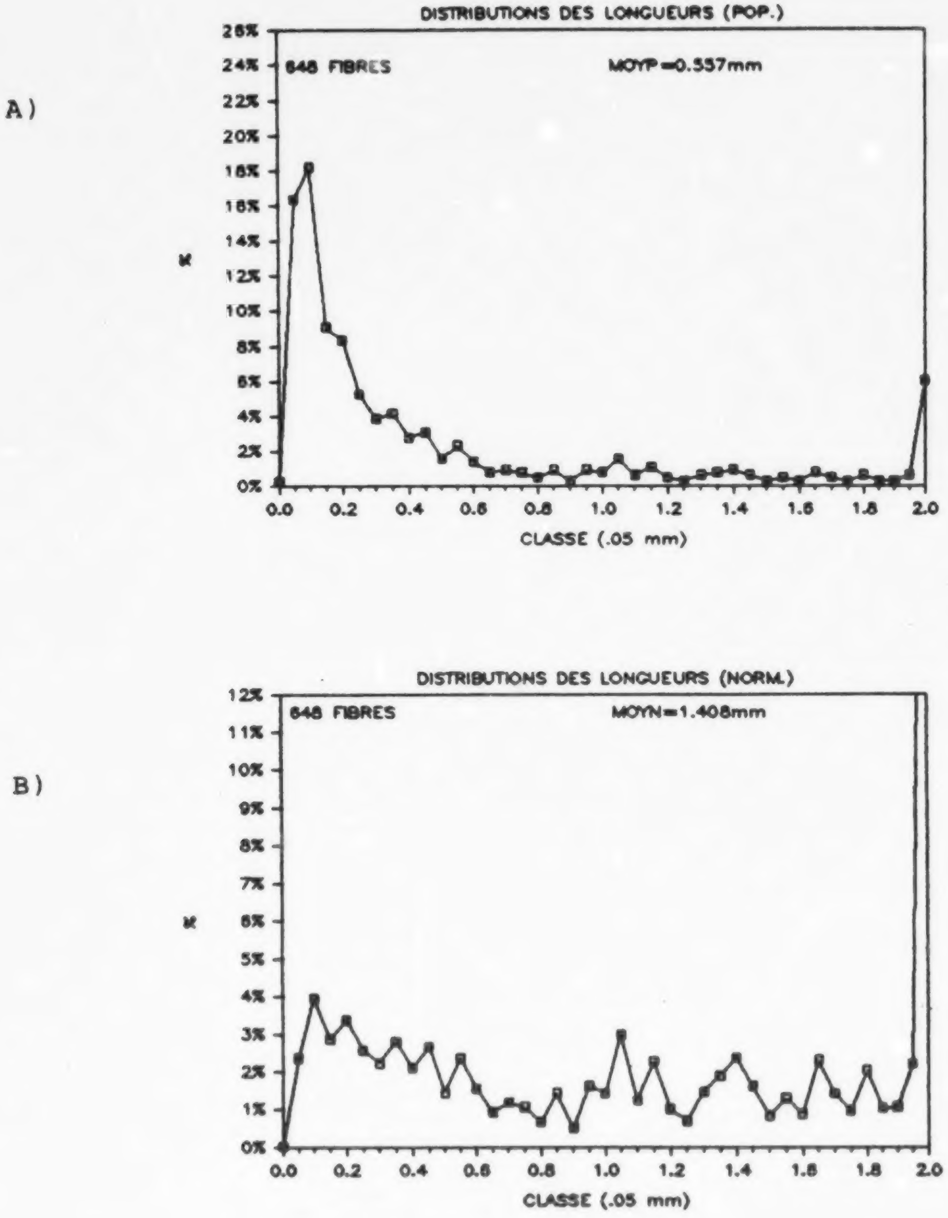


FIG. VIII.3i) Length distributions of AX Pallmann opened fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

QUEBEC DRY-MILLED 4T FIBERS (Open)

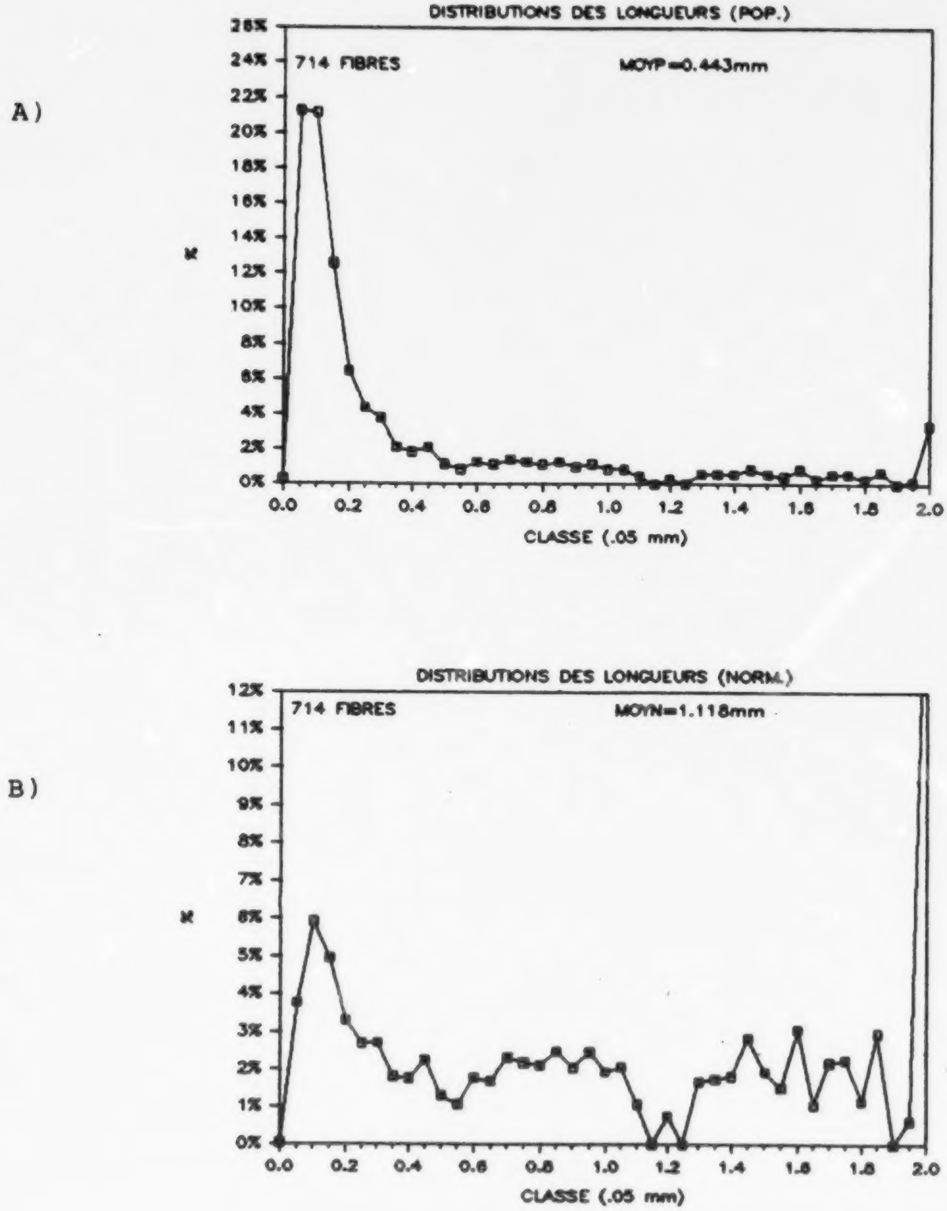


FIG. VIII.3 j) Length distributions of 4T Pallmann opened fibers obtained through optical microscopy, by .05 mm classes.

A) Population
B) Normalized

Examination of the $\bar{\ell}$ and σ_ℓ data, and comparisons of these with corresponding data for the bulk fiber products (Table VIII.1) show the following:

- In most cases, the average fiber lengths found before and after Pallmann opening agree within 20%.
- With wet-milled fiber products (Cassiar or Woodsreef), the mean fiber lengths and standard deviations both decrease slightly upon Pallmann treatment; the resulting fibers are thus slightly (10-20%) shorter, but they are more homogeneous in length (lower σ_ℓ).
- The length homogeneity of the Cassiar wet-milled fibers is again greater than for all other wet-milled products.
- In most cases, with dry-milled fibers (Cassiar or JM), the mean lengths and the standard deviations apparently increase upon Pallmann opening. This unexpected effect may be due to the complex shapes of the dry-milled fibers which complicate the collection of statistically valid data. Alternately it could simply reflect a larger number of long fibers resulting from splitting of the large bundles.

b) Fiber diameter distributions

The distributions (population and normalized) of fiber diameters for Pallmann opened fibers are illustrated in Fig. VIII.4a-j; the computed mean diameter (d) and standard deviation values (σ_d) are given in Table VIII.4.

CASSIAR WET-MILLED S FIBERS (Open)

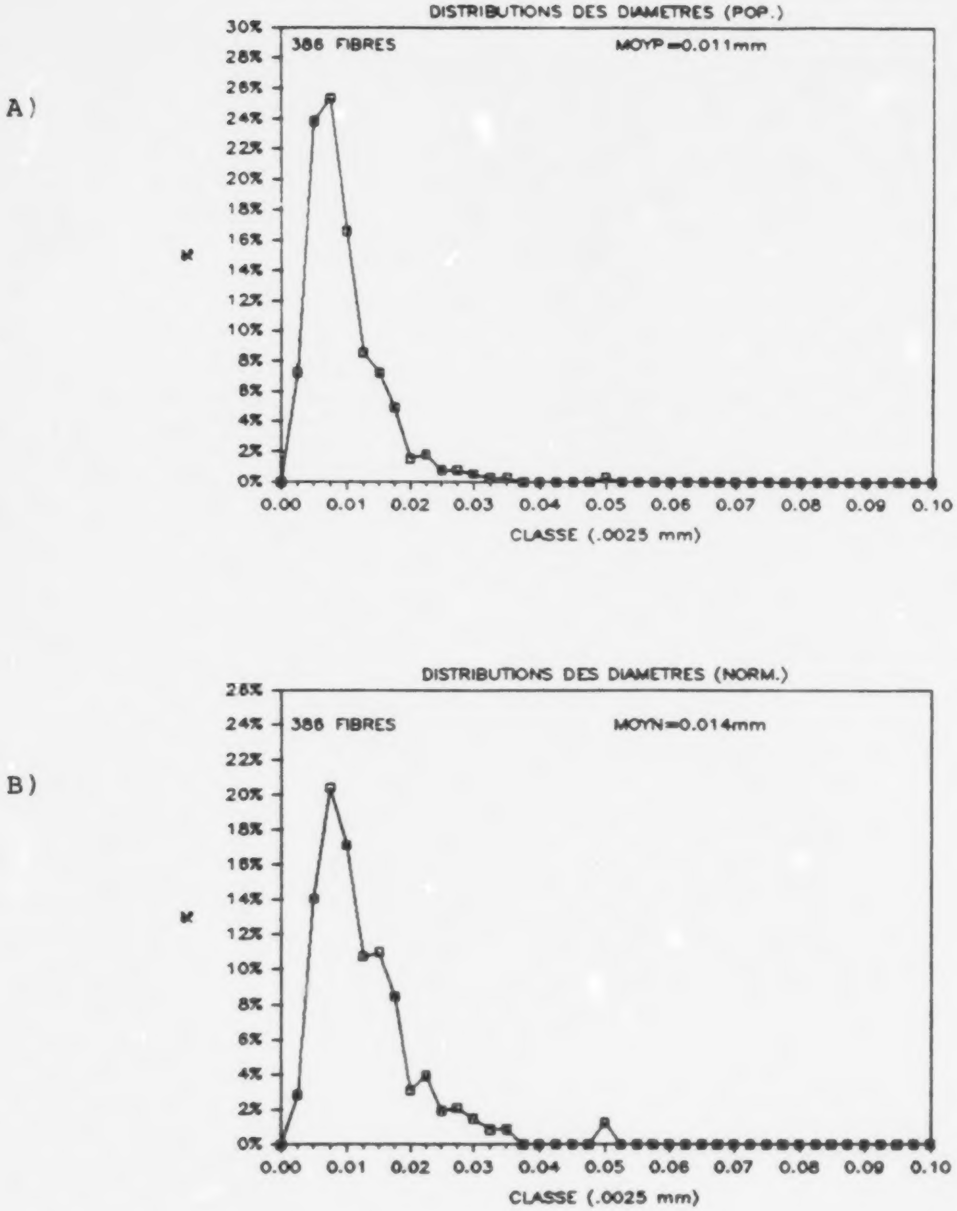


FIG. VIII.4a) Diameter distributions of S Pallmann opened fibers obtained through optical microscopy, by .0025 mm classes

A) Population
B) Normalized

(Class labels on X-coordinates give the lower limit of each class.)

CASSIAR WET-MILLED M FIBERS (Open)

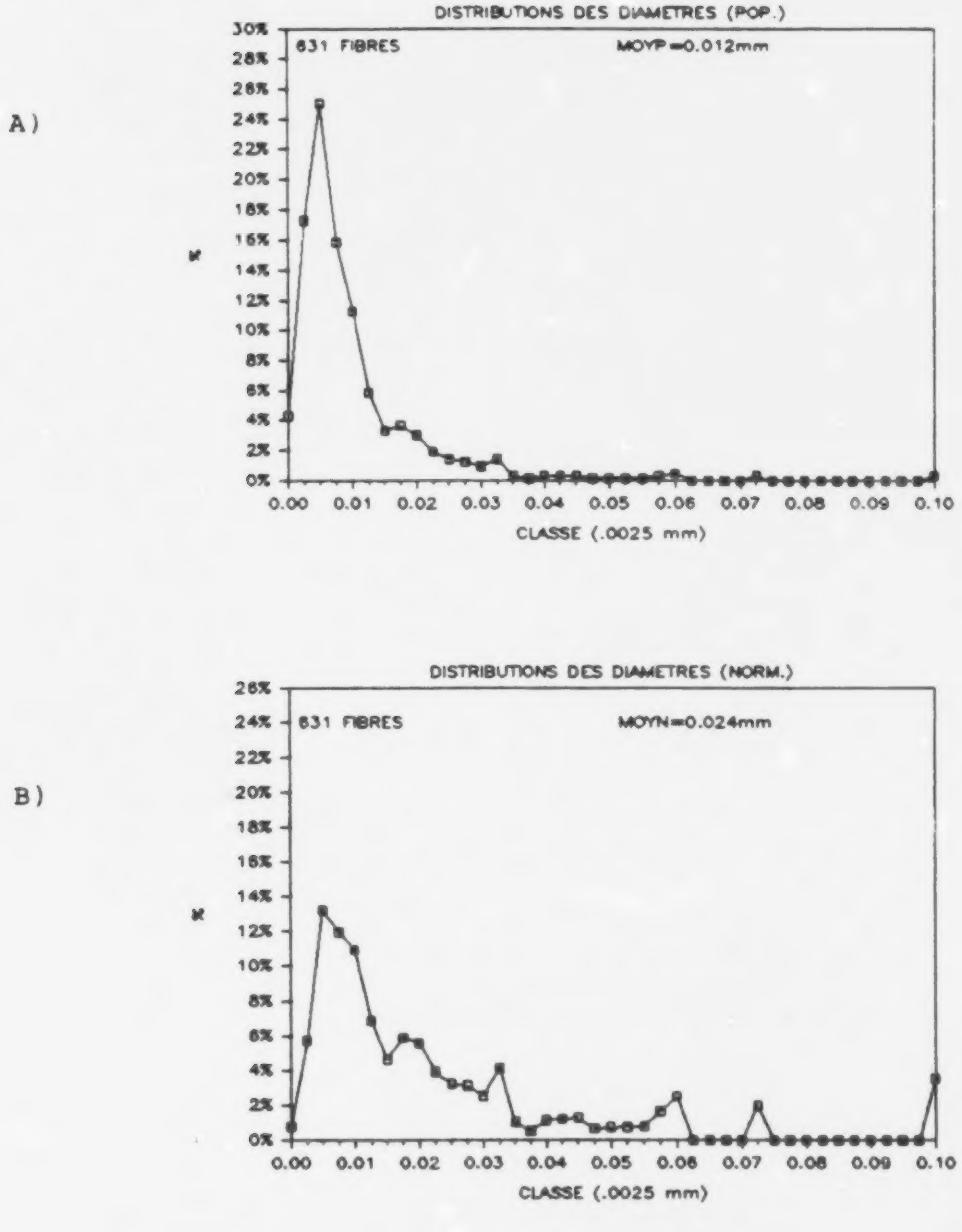


FIG. VIII.4b) Diameter distributions of M Pallmann opened fibers obtained through optical microscopy, by .0025 mm classes

A) Population
B) Normalized

CASSIAR WET-MILLED L FIBERS (Open)

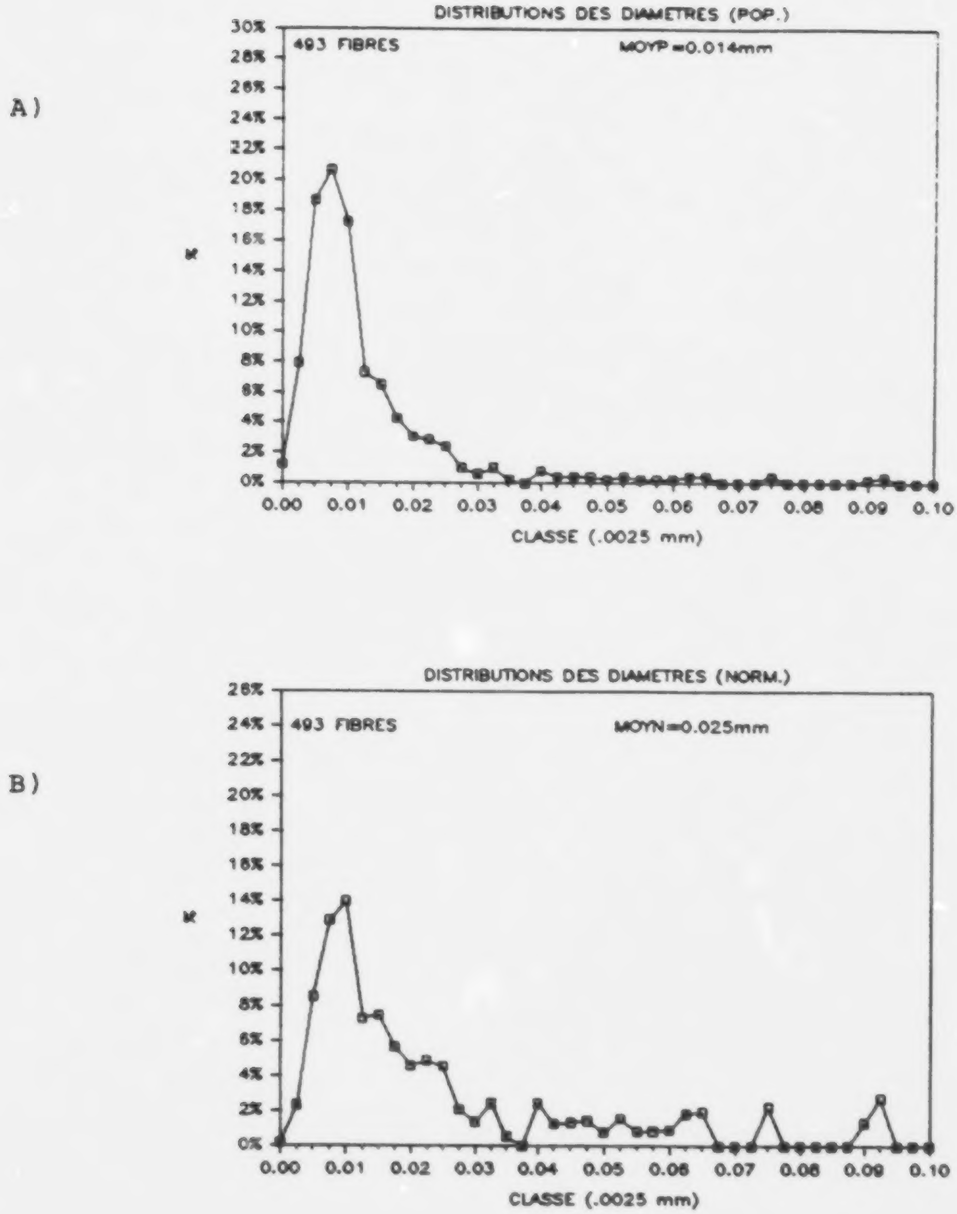


FIG. VIII.4c) Diameter distributions of L Pallmann opened fibers obtained through optical microscopy, by .0025 mm classes

A) Population
B) Normalized

CASSIAR WET-MILLED XL FIBERS (Open)

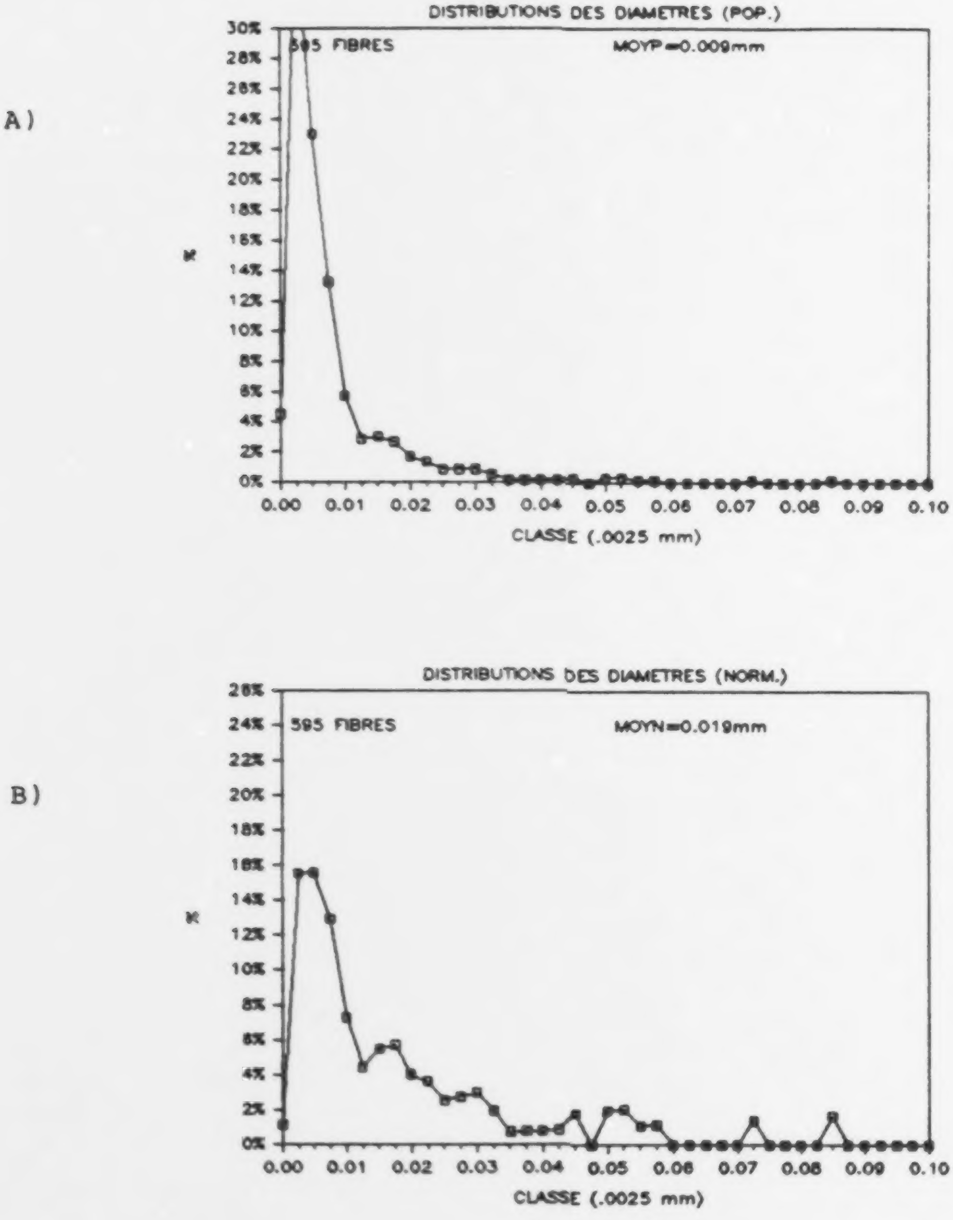


FIG. VIII.4d) Diameter distributions of XL Pallmann opened fibers obtained through optical microscopy, by .0025 mm classes

A) Population
B) Normalized

CASSIAR WET-MILLED FEED FIBERS (Open)

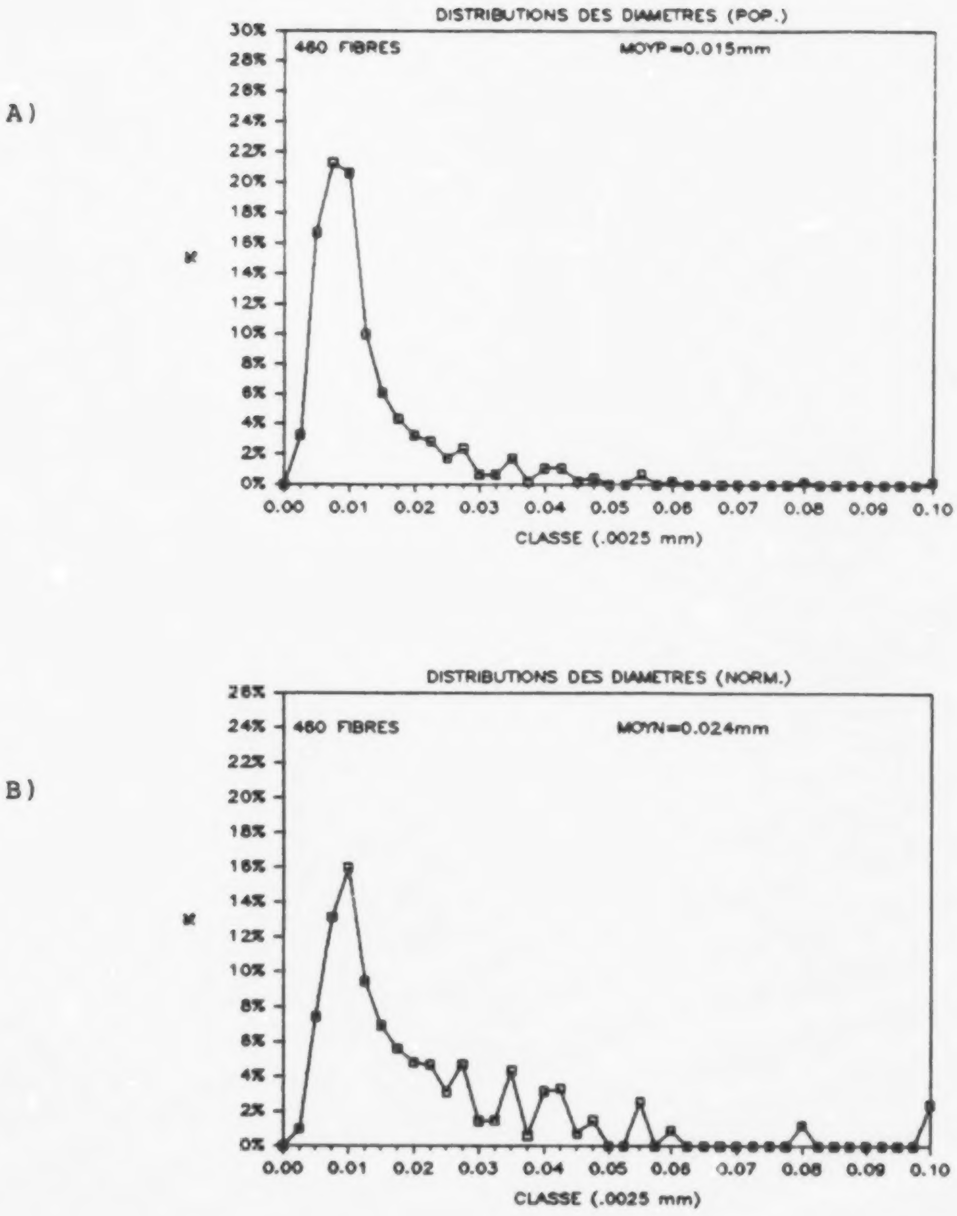


FIG. VIII.4e) Diameter distributions of FEED Pallmann opened fibers obtained through optical microscopy, by .0025 mm classes

A) Population
B) Normalized

WOODSREEF WET-MILLED 10-MESH FIBERS (Open)

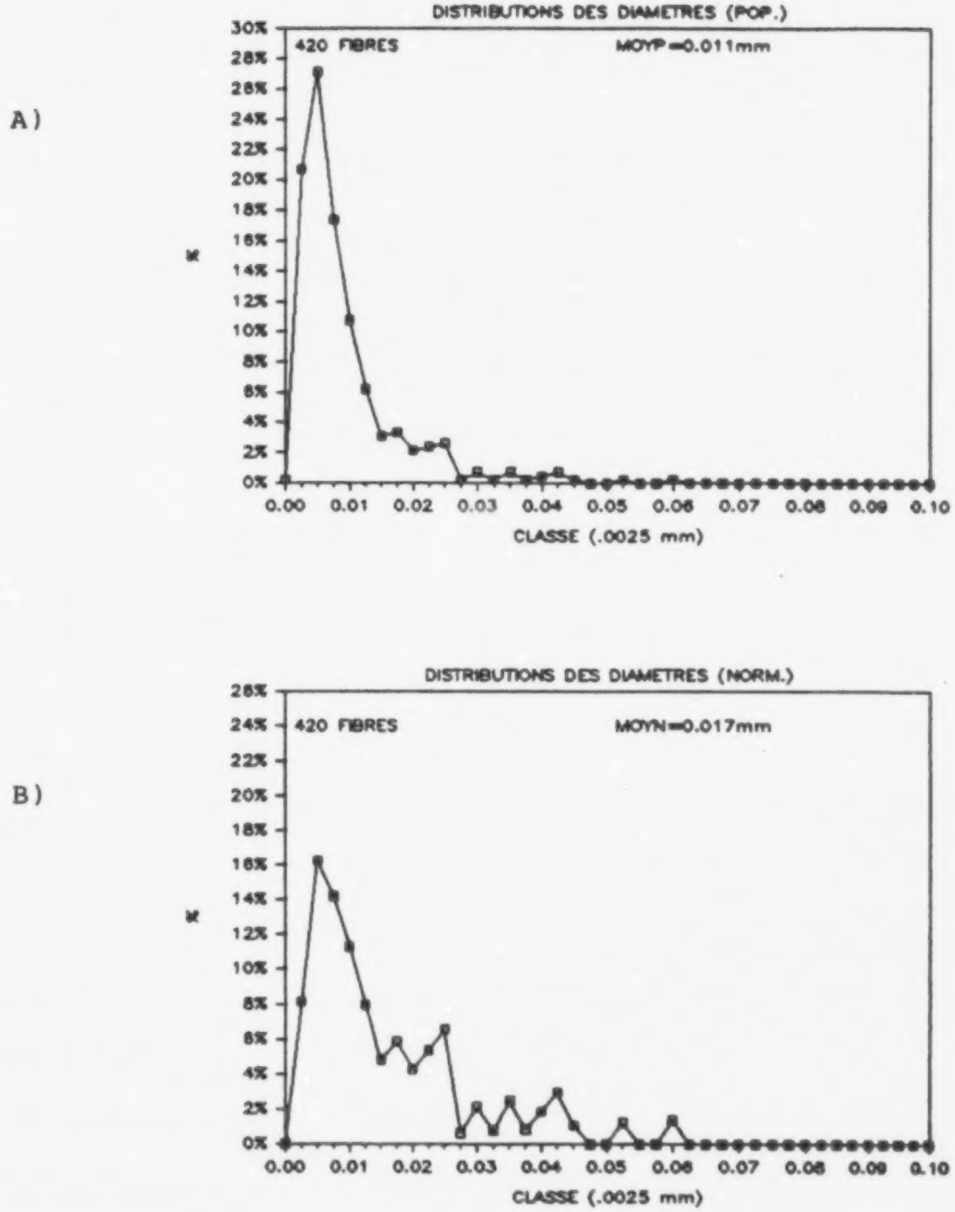


FIG. VIII.4f) Diameter distributions of Woodsreef 10-mesh Pallmann opened fibers obtained through optical microscopy, by .0025 mm classes

A) Population
B) Normalized

CASSIAR DRY-MILLED AZ FIBERS (Open)

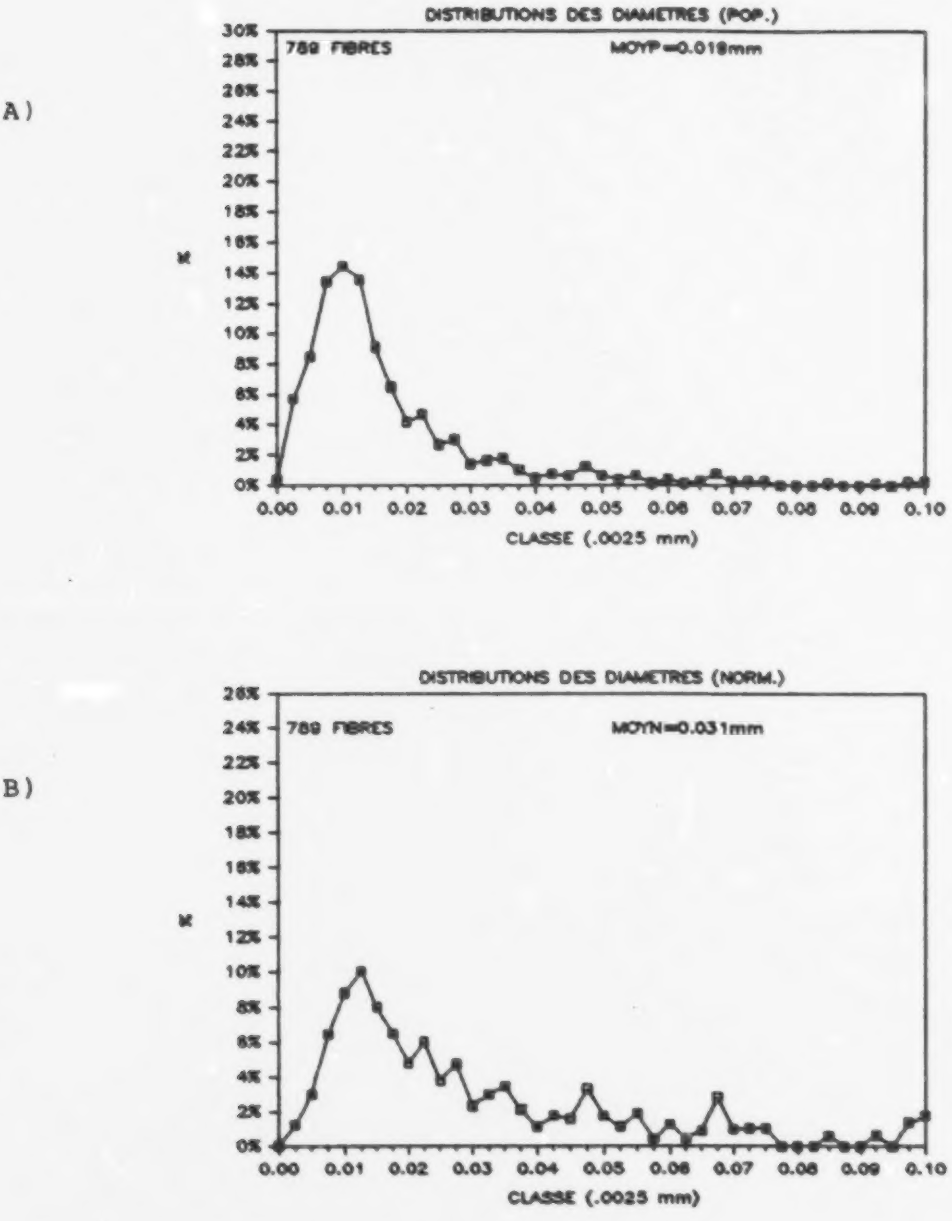


FIG. VIII.4g) Diameter distributions of AZ Pallmann opened fibers obtained through optical microscopy, by .0025 mm classes

A) Population
B) Normalized

CASSIAR DRY-MILLED AY FIBERS (Open)

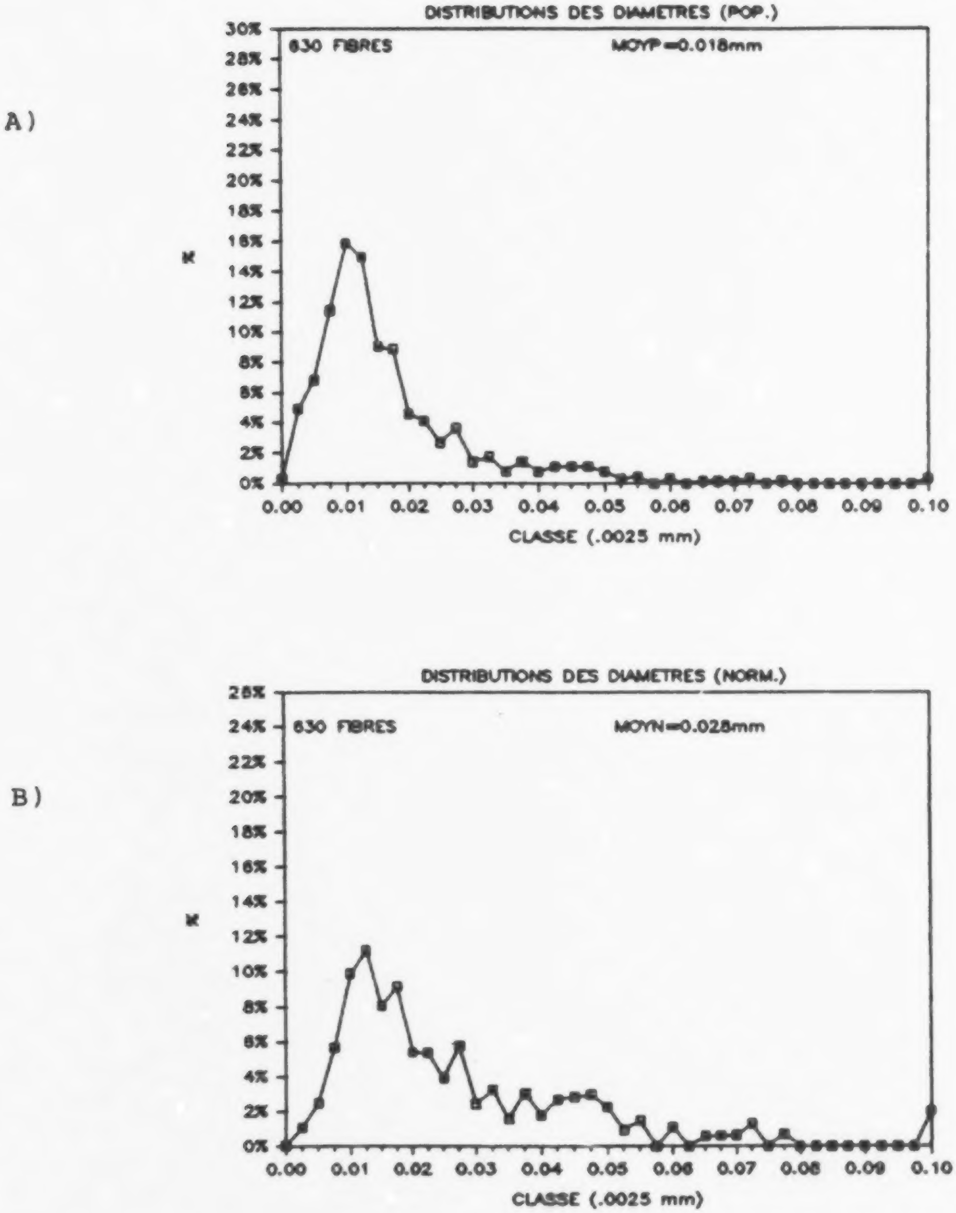


FIG. VIII.4h) Diameter distributions of AY Pallmann opened fibers obtained through optical microscopy, by .0025 mm classes

A) Population
B) Normalized

CASSIAR DRY-MILLED AX FIBERS (Open)

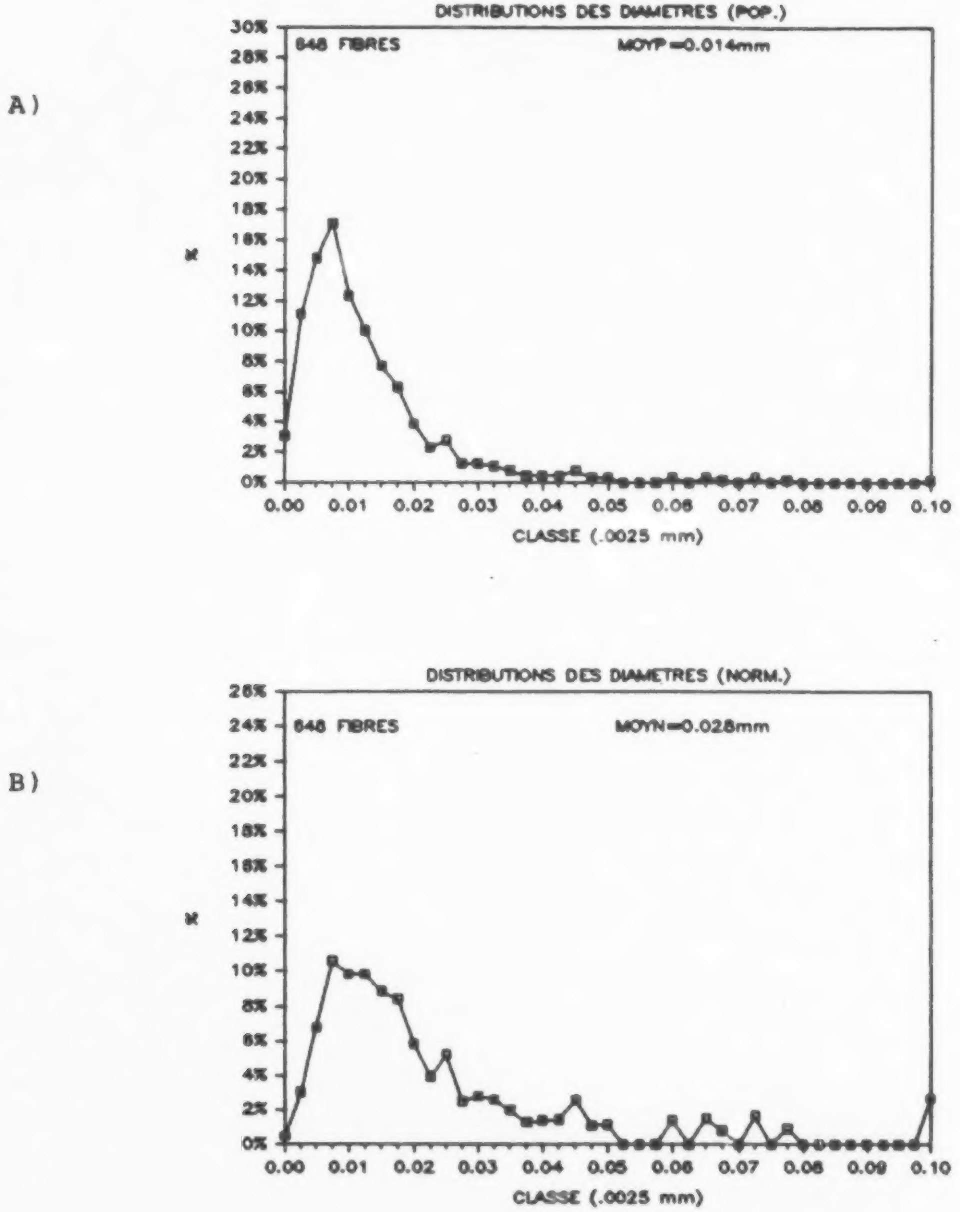


FIG. VIII.4i) Diameter distributions of AX Pallmann opened fibers obtained through optical microscopy, by .0025 mm classes

A) Population
B) Normalized

QUEBEC DRY-MILLED 4T FIBERS (Open)

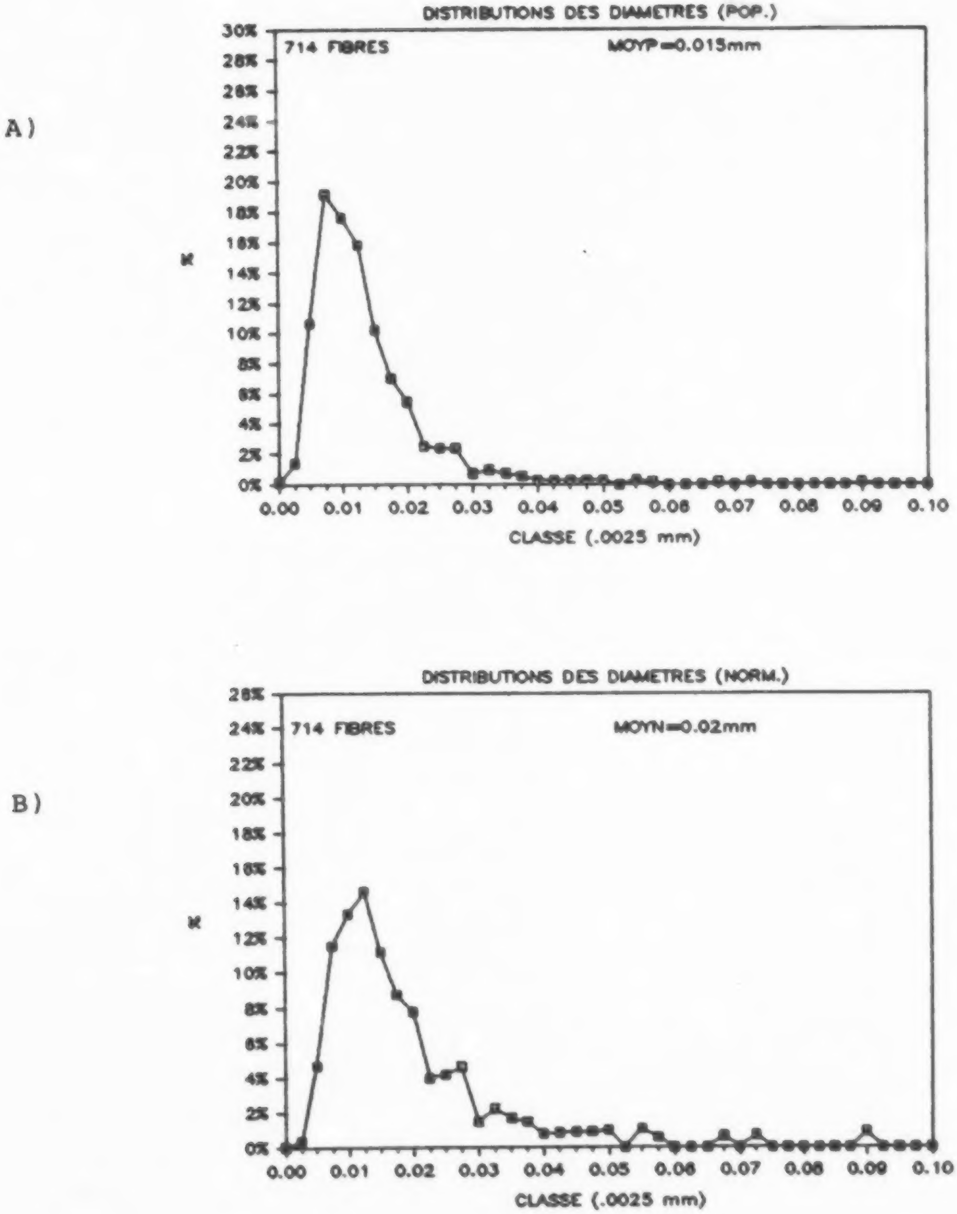


FIG. VIII.4j) Diameter distributions of 4T Pallmann opened fibers obtained through optical microscopy, by .0025 mm classes

A) Population
B) Normalized

TABLE VIII.4: Mean diameter ($\bar{d}$) and standard deviations (σ_d) from OM diameter distributions of Pallmann opened fibers

Sample		No. of fibers	Population (mm) $\bar{d}$	σ_d	Normalized (mm) $\bar{d}$
Wet-milled	Cassiar S	386	.011	(.006)	.014
"	Cassiar M	631	.012	(.012)	.024
"	Cassiar L	493	.014	(.013)	.025
"	Cassiar XL	595	.009	(.009)	.019
"	Cassiar FEED	460	.015	(.012)	.024
"	Woodsreef 10-Mesh	630	.011	(.008)	.017
Dry-milled	Cassiar AZ	789	.019	(.015)	.031
"	Cassiar AY	630	.018	(.013)	.028
"	Cassiar AX	648	.014	(.014)	.028
"	Quebec 4T	714	.015	(.009)	.020

The trends observed in the $\bar{d}$ and σ_d results and comparisons among the data for bulk and opened fibers lead to the following conclusions:

- With all types of fibers examined, the Pallmann opening treatment leads to only minor reductions of the average fiber diameters.

- As reflected by σ_d values (or σ_d/d), the homogeneity of fiber diameters is improved for most fibers following Pallmann opening mainly as result of opening of the larger fiber bundles; this is particularly noteworthy with the wet-milled FEED fibers suggesting that the larger fibers in wet-milled products are easier to open.

- After Pallmann opening, the average diameters of the wet-milled classified Cassiar (S, M, L, XL) fibers and Woodsreef 10-mesh fibers fall within a narrow range of values around 12 μm; the mean diameters of dry-milled fibers are significantly larger, falling in a narrow range around 17 μm.

VIII.3 - Consequences of Pallmann opening on fiber size and size distributions on Cassiar wet-milled fibers

The various points noted above were concluded from examination of mean values of fiber lengths and diameter. To further understand how the various classes of fiber lengths (or diameters) are affected by Pallmann opening, we plotted differential histograms showing directly for each class in the distributions (ℓ or d) the changes found upon Pallmann treatment. This procedure was carried out only for the Cassiar wet-milled fibers as is most relevant to the present study. The corresponding figures are collected in Appendix G (Fig. G-1(a-e) and Fig. G-2(a-e)); clearly, the differential histograms bear large uncertainties, but some trends were found significant.

a) Changes in fiber length distributions

Examination of the differential histograms of wet-milled fiber lengths (Appendix G, Fig. G-1(a-e)) supports the following statements:

- Pallmann opening has no marked influence on fiber lengths in the S-grade.
- With M and L grades, there appears a minor increase of populations in the short fiber classes with a corresponding decrease in the intermediate length classes.
- No significant trend is detected in length changes for the XL fibers.
- The FEED fibers exhibit a weak systematic decrease in the long fiber classes and a corresponding increase in the short fiber classes.

The changes noted here lie close to the resolution and statistical validity of our methods, but they systematically show that only minor reductions of fiber lengths occur in the Pallmann opening of the wet-milled fibers. Except, possibly, with the bulk FEED fibers, the length reduction observed in Pallmann opening should bear no measurable consequences in the fiber strength properties.

b) Changes in fiber diameter distributions

The changes in diameter distributions of the Cassiar wet-milled fibers following Pallmann opening can be vizualized from the differential population histograms (Appendix G, Fig. G.1(a-e)); these show that:

- With the S fibers, a systematic increase is noted in the smaller diameter classes, with corresponding decrease in the intermediate classes.
- With M, L and XL grades, changes in the distribution appear too erratic to be interpreted meaningfully.[1]
- With the FEED fibers, a substantial increase in the small diameter classes is found with a systematic decrease in larger diameter classes.

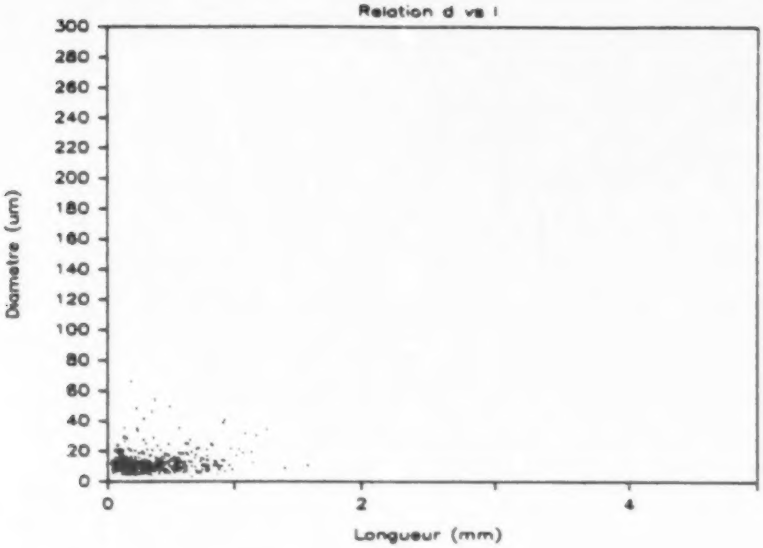
The consequences of Pallmann opening on fiber lengths and diameters of the various wet- and dry-milled products may also be illustrated through the ℓ-d correlation plots shown in Fig. VIII.5(a-i). In these plots, each point represents a length and diameter measured for individual fibers by optical microscopy; all plots are shown at the same scale to facilitate visual examination.

---

[1] As noted in previous sections, the diameter measurement and interpretation are complicated by the various physical effects produced during Pallmann opening, namely the appearance of "bruises", "kinks" and "splittings" of the fibers which tend to increase their apparent diameters.

CASSIAR WET-MILLED S FIBERS

A)



B)

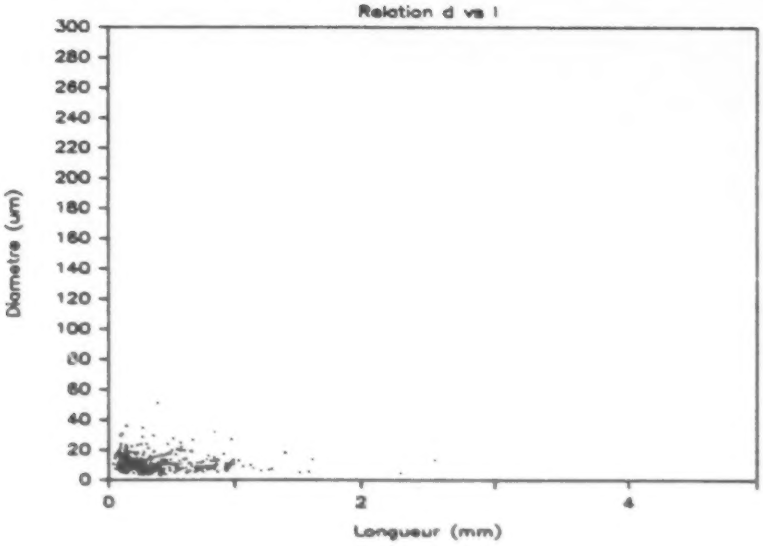


FIG. VIII.5a) Length-diameter correlation plots obtained from optical microscopy measurements on S fibers; each point represents the length and diameter of an individual fiber

A) Bulk
B) Pallmann opened

CASSIAR WET-MILLED M FIBERS

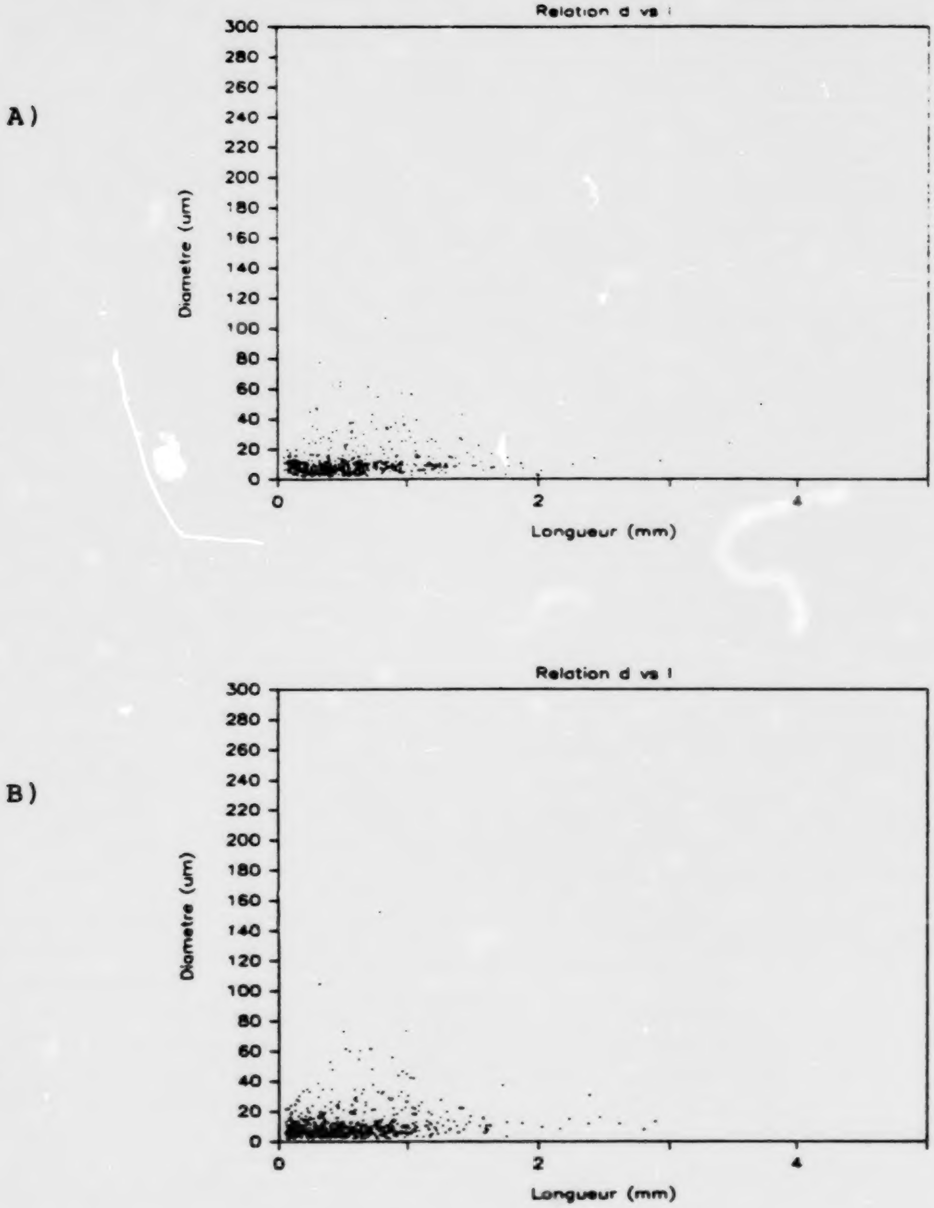


FIG. VIII.5b) Length-diameter correlation plots obtained from optical microscopy measurements on M fibers; each point represents the length and diameter of an individual fiber

A) Bulk
B) Pallmann opened

CASSIAR WET-MILLED L FIBERS

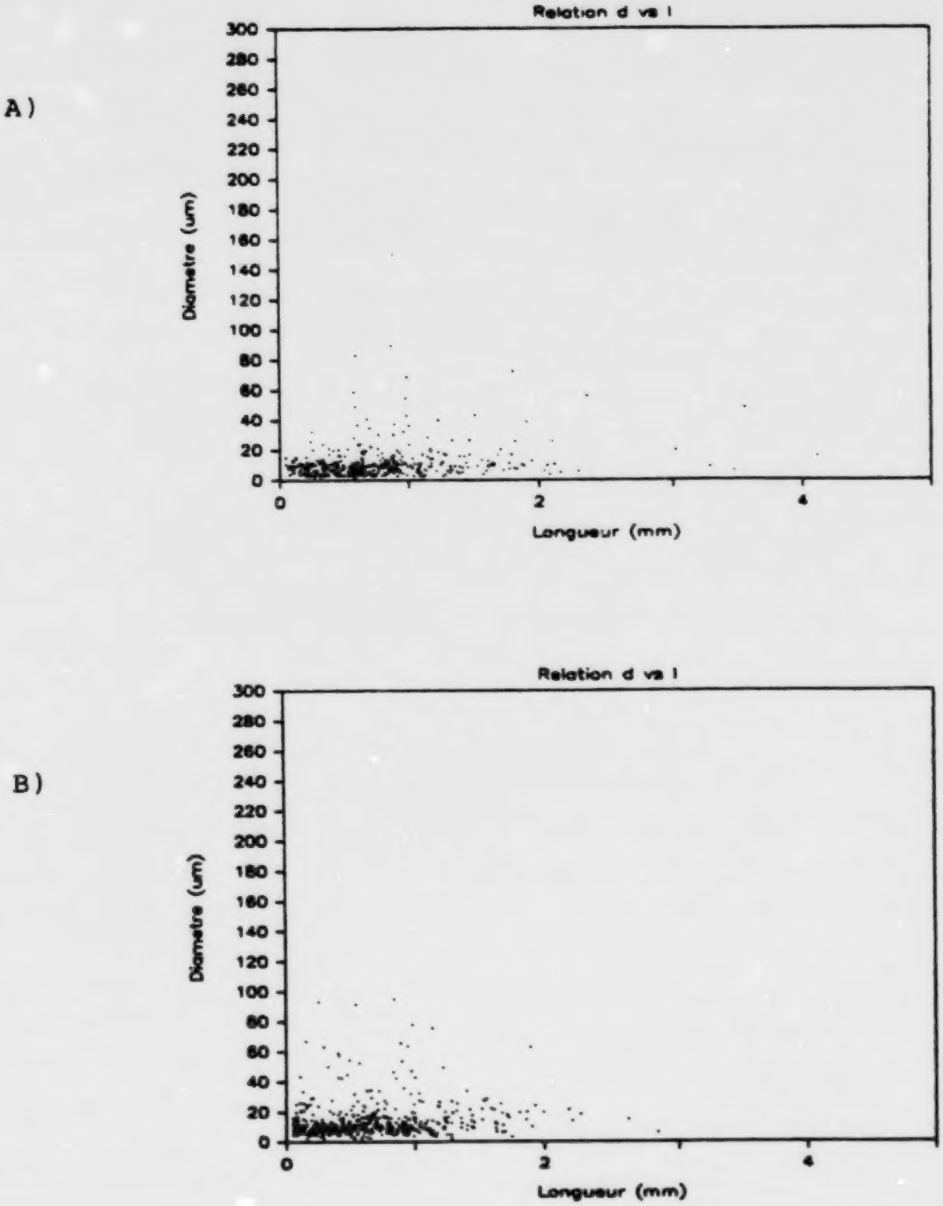


FIG. VIII.5c) Length-diameter correlation plots obtained from optical microscopy measurements on L fibers; each point represents the length and diameter of an individual fiber

A) Bulk
B) Pallmann opened

CASSIAR WET-MILLED XL FIBERS

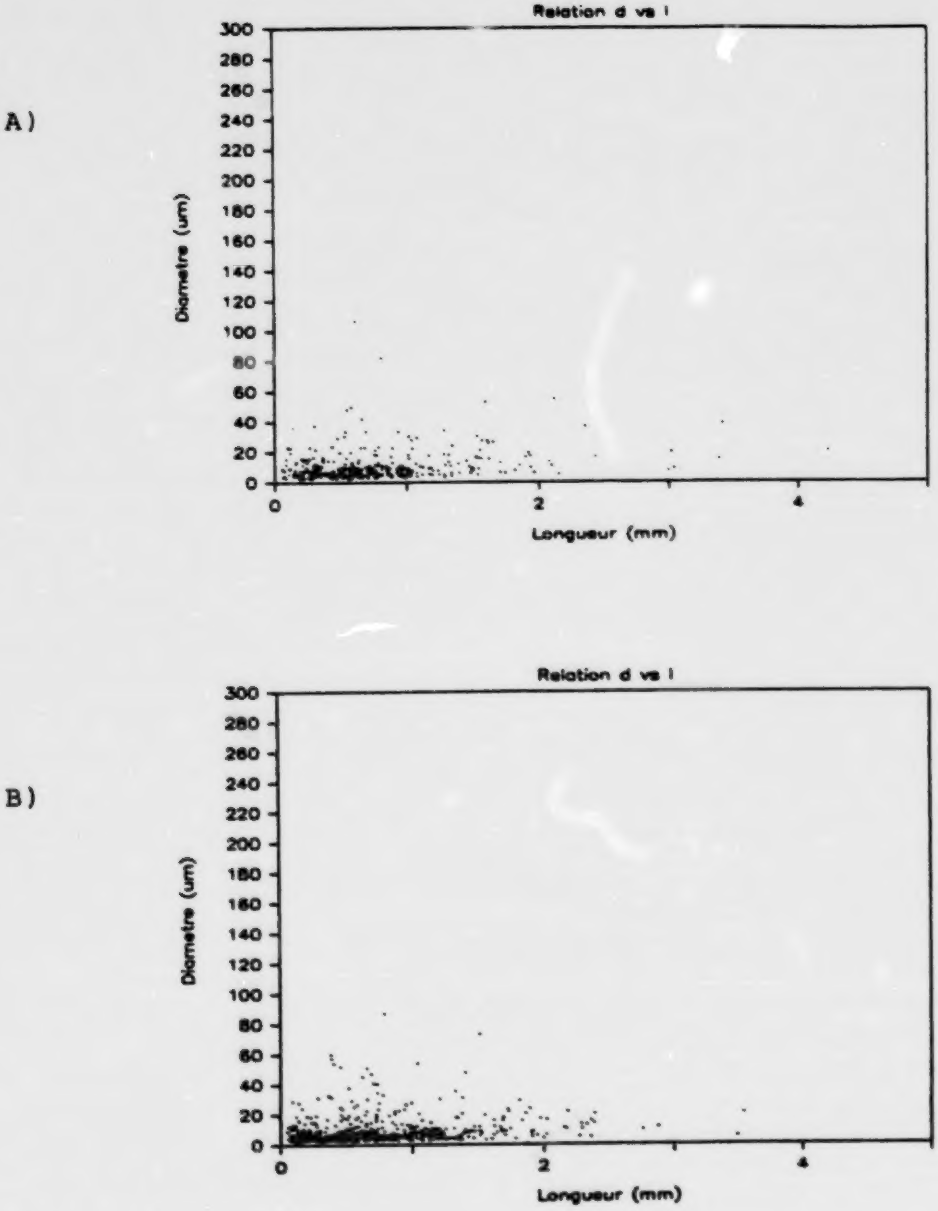


FIG. VIII.5d) Length-diameter correlation plots obtained from optical microscopy measurements on XL fibers; each point represents the length and diameter of an individual fiber

A) Bulk
B) Pallmann opened

CASSIAR WET-MILLED FEED FIBERS

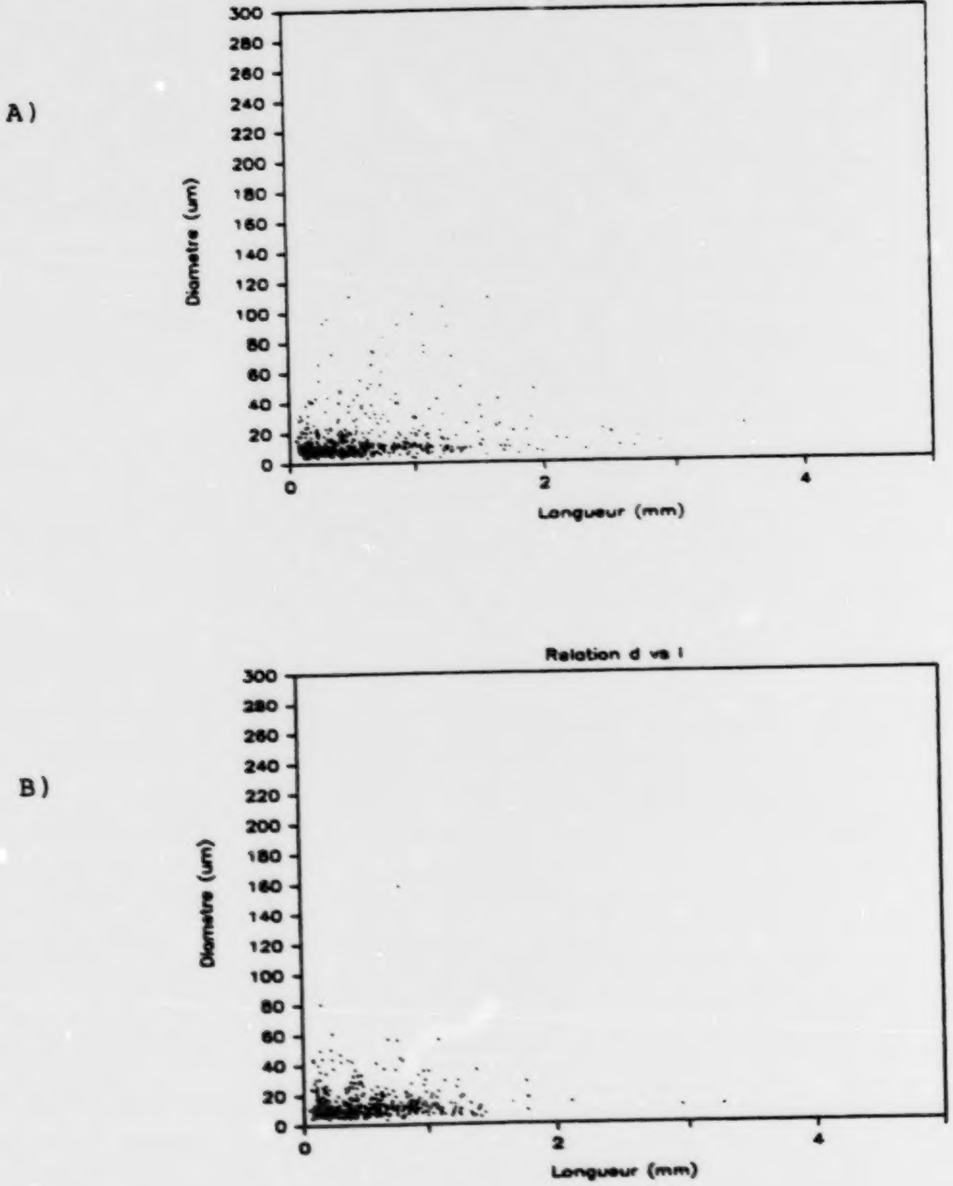


FIG. VIII.5e) Length-diameter correlation plots obtained from optical microscopy measurements on FEED fibers; each point represents the length and diameter of an individual fiber

A) Bulk
B) Pallmann opened

WOODSREEF WET-MILLED 10-MESH FIBERS

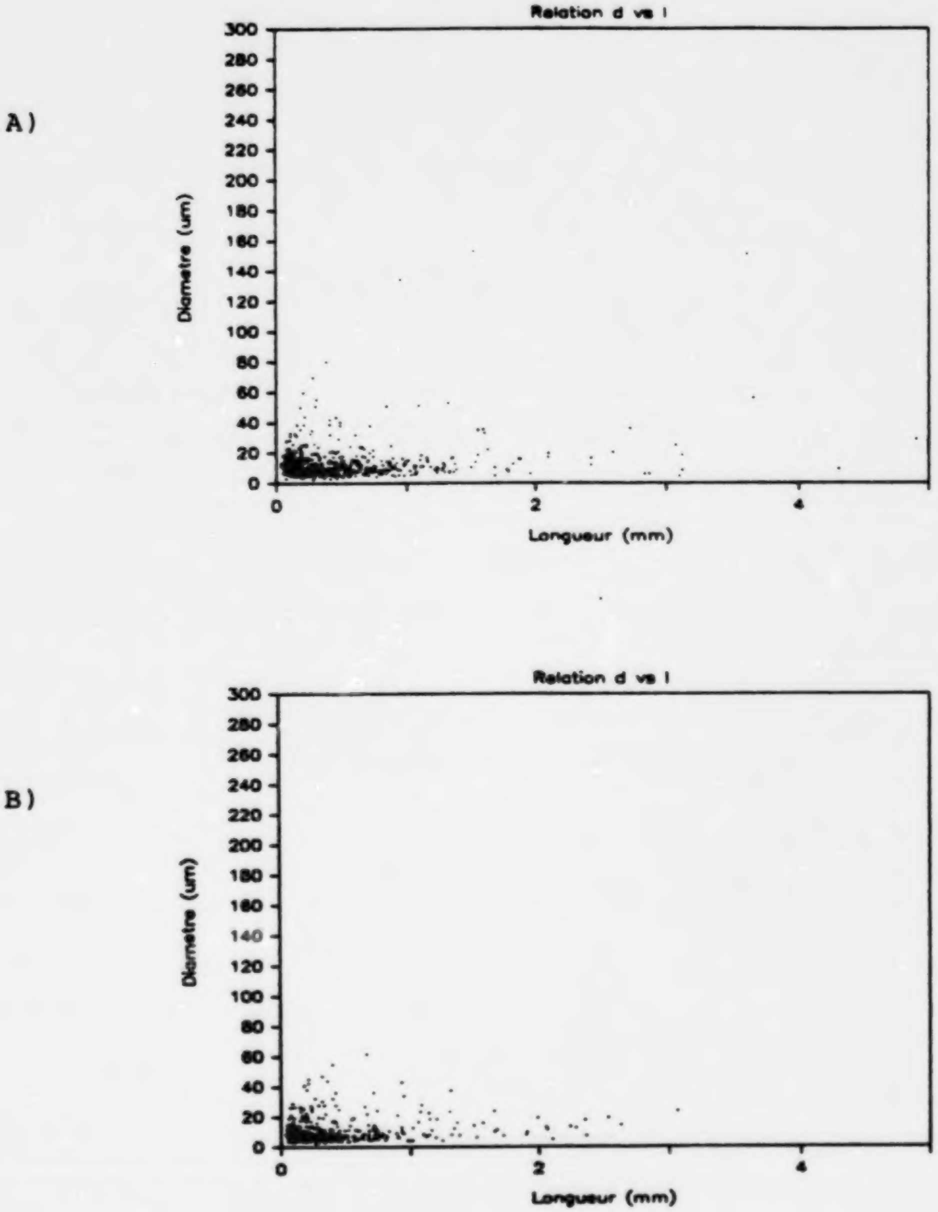


FIG. VIII.5f) Length-diameter correlation plots obtained from optical microscopy measurements on Woodsreef 10-mesh fibers; each point represents the length and diameter of an individual fiber

A) Bulk
B) Pallmann opened

CASSIAR DRY-MILLED AZ FIBERS

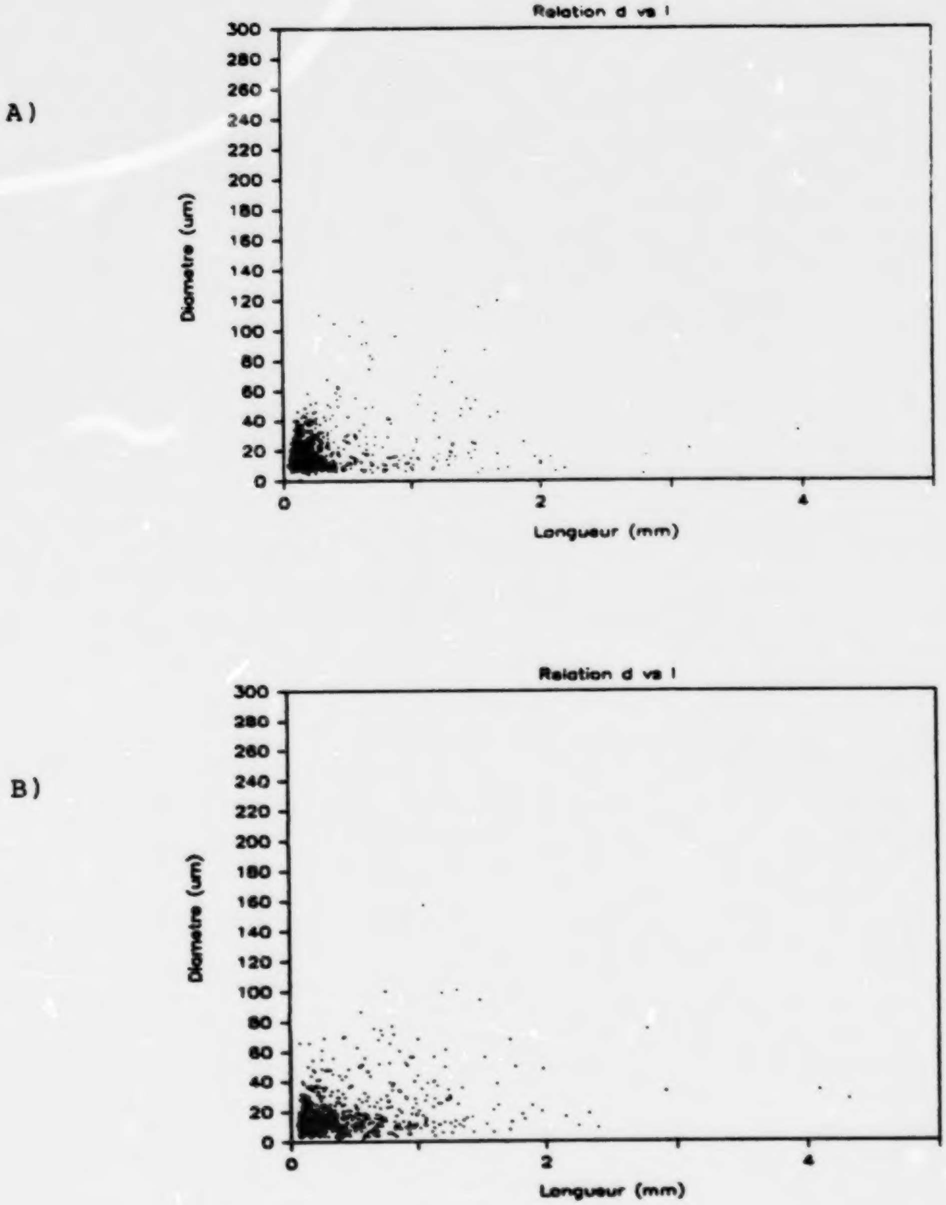


FIG. VIII.5g) Length-diameter correlation plots obtained from optical microscopy measurements on AZ fibers; each point represents the length and diameter of an individual fiber

A) Bulk
B) Pallmann opened

CASSIAR DRY-MILLED AY FIBERS

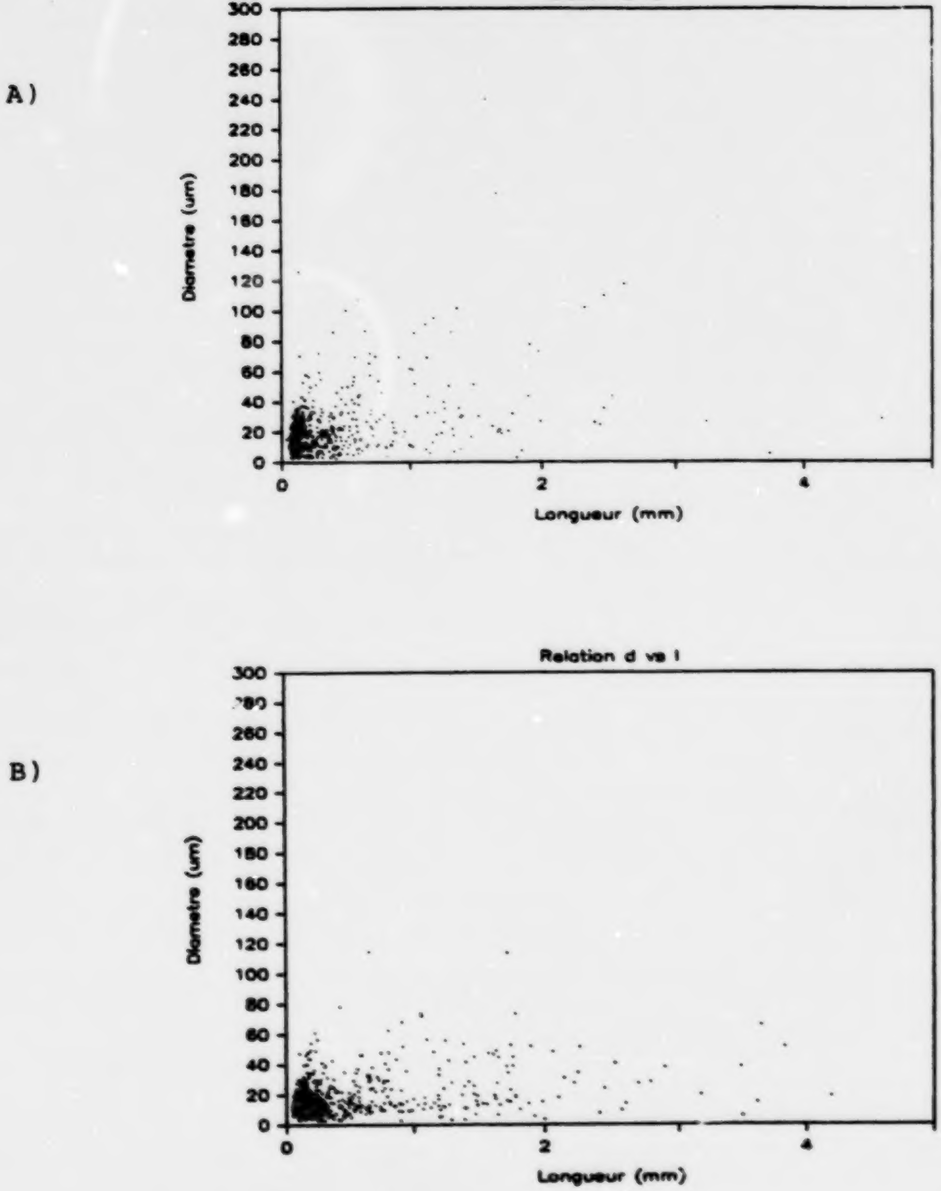
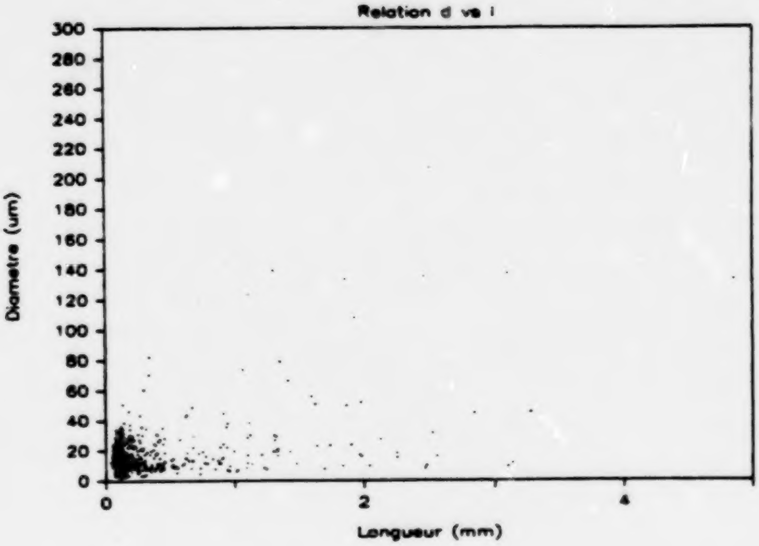


FIG. VIII.5h) Length-diameter correlation plots obtained from optical microscopy measurements on AY fibers; each point represents the length and diameter of an individual fiber

A) Bulk
B) Pallmann opened

CASSIAR DRY-MILLED AX FIBERS

A)



B)

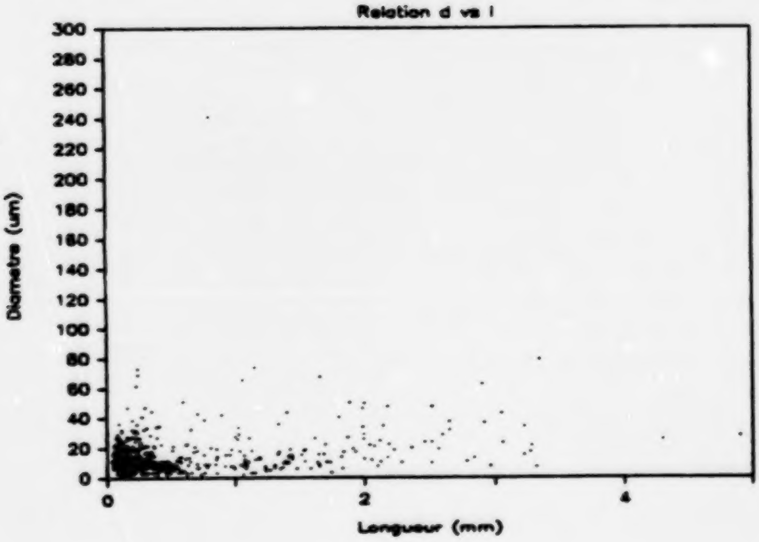


FIG. VIII.5i) Length-diameter correlation plots obtained from optical microscopy measurements on AX fibers; each point represents the length and diameter of an individual fiber

A) Bulk
B) Pallmann opened

Comparing the two correlation plots reproduced in each figure (upper plot: bulk fibers; lower plot: Pallmann opened fibers), it is readily seen that the main consequence of Pallmann opening is a substantial reduction in the numbers of large diameter bundles, i.e., fibers with diameters in excess of 30-40 µm. With wet-milled fibers, the reduction is particularly evident for the bulk Cassiar FEED product and with the Woodsreef 10-mesh product. The same effect is also very pronounced with dry-milled fibers.

Examination of fiber length changes from these correlation plots also points to minor reductions in the number of the longer fibers upon Pallmann opening; however, because of the length dispersity, the effect is not as evident as it was for diameters.

Finally, the ℓ-d correlation plots nicely illustrate the greater homogeneity of Pallmann opened fibers, i.e., the data points are more closely grouped for opened fibers than for bulk fibers.

VIII.4 - Length distributions using the Kajaani FS-100 analyser

Fiber length distributions were obtained with a Kajaani FS-100 Fiber Length Analyser for most of the fiber products studied here, both before and after Pallmann opening.[1] The results are reported below and compared to distributions obtained by optical microscopy after matching of the OM length classes to those used in the Kajaani FS-100 instrument (0.2 mm increments).

---

[1] With fiber products containing excessive crude fibers (larger bundles) frequent clogging of the Kajaani capillary made the measurements more difficult; hence no results are reported here for the Quebec 4T fibers.

a) Bulk fibers

We illustrate in Fig. VIII.6a, b, c, typical fiber length distributions obtained with the Kajaani FS-100 for the Cassiar wet-milled S, XL and AY fibers. Each figure gives the results for four different runs; the weak scattering of the data clearly confirms excellent reproducibility. The distributions obtained for the Cassiar wet-milled S, M, L and XL fibers and the Woodsreef 10-mesh fibers are collected in Fig. VIII.7(a, b); similarly, the length distributions for the bulk dry-milled fibers AZ, AY and AX are illustrated in Fig. VIII.7c.

Finally, the mean lengths ($\bar{\ell}$) and standard deviations (σ_ℓ) obtained from the Kajaani distributions are collected in Table VIII.5, together with data obtained on the Pallmann opened fibers. Detailed comparison of the Kajaani data with those obtained via optical microscopy will be carried out in section XI.1.

TABLE VIII.5: Mean lengths (ℓ) of wet- and dry-milled fibers from Cassiar and wet-milled fibers from Woodsreef obtained with a Kajaani Fiber Length Analyser (FS-100)

Sample	Population $\bar{\ell}$ (mm)	Normalized $\bar{\ell}$ (mm)
Wet-milled		
Cassiar S	.248	.472
" M	.468	.750
" L	.564	.970
" XL	.572	.940
Woodsreef 10-mesh	.232	.440
Dry-milled		
Cassiar AZ	.244	.548
" AY	.268	.622
" AX	.288	.740

The mean length values computed by our Kajaani FS-100 instrument is incorrect; values are between 25 and 50% shorter than they should be based on the distributions.

CASSIAR WET-MILLED S FIBERS (Bulk)

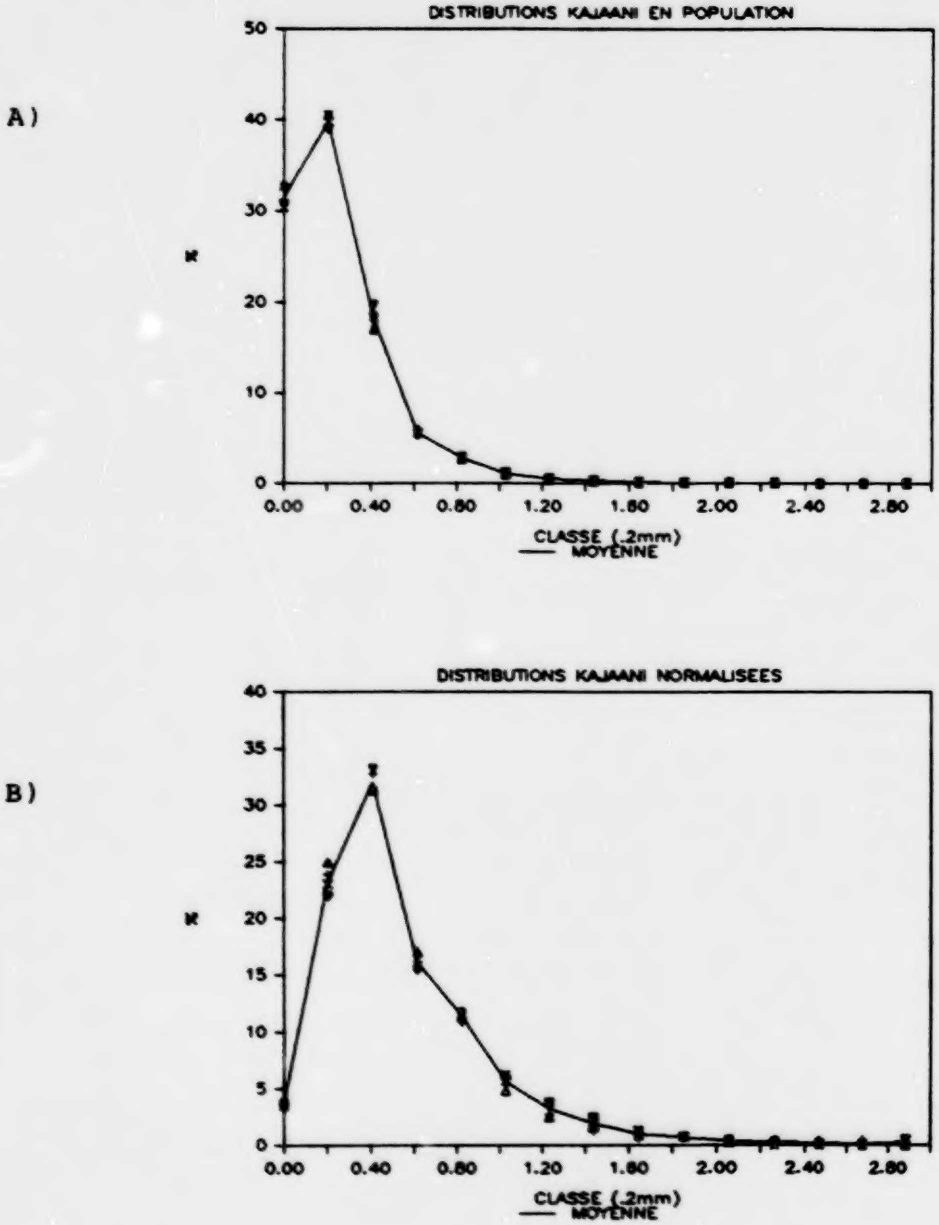


FIG. VIII.6a) Length distribution of wet-milled S fibers obtained from a FS-100 KAJAANI Fiber Length Analyser, by .21 mm classes. Symbols are related to single experiments and lines are the resulting means.

A) Population
B) Normalized

(Class labels on X-coordinates give the lower limit of each class.)

CASSIAR WET-MILLED XL FIBERS (Bulk)

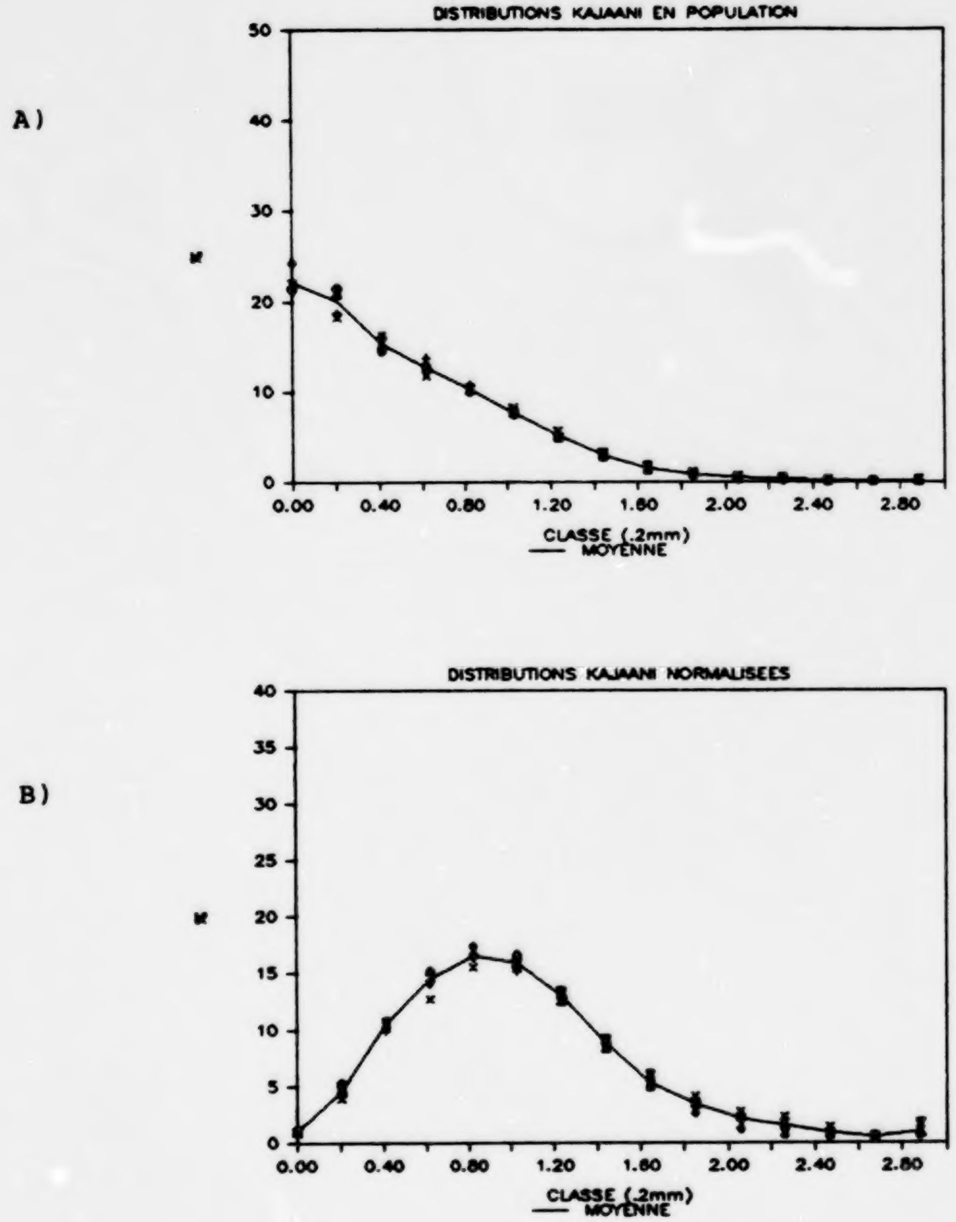
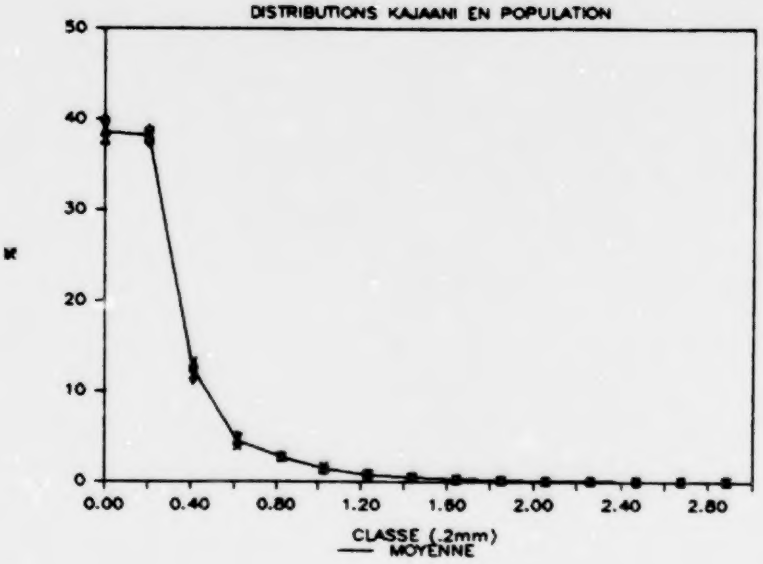


FIG. VIII.6b) Length distribution of wet-milled XL fibers obtained from a FS-100 KAJAANI Fiber Length Analyser, by .21 mm classes. Symbols are related to single experiments and lines are the resulting means.

A) Population
B) Normalized

CASSIAR DRY-MILLED AY FIBERS (Bulk)

A)



B)

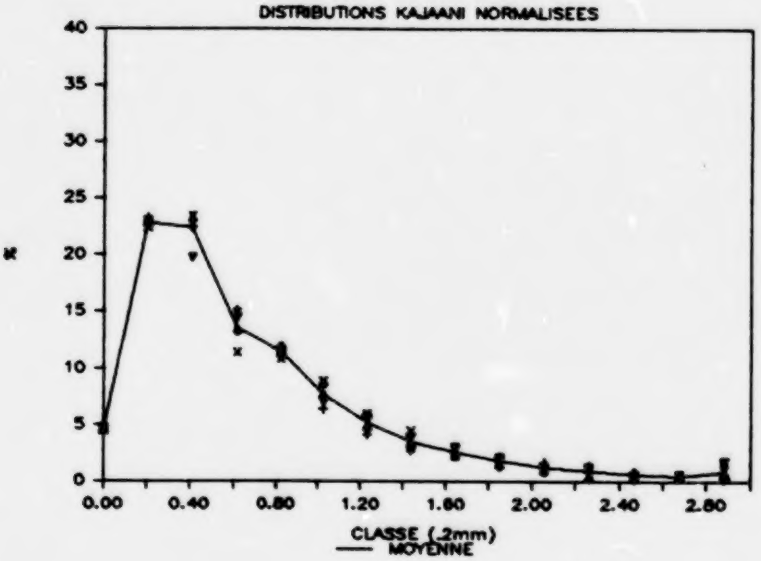


FIG. VIII.6c) Length distribution of dry-milled AY fibers obtained from a FS-100 KAJAANI Fiber Length Analyser, by .21 mm classes. Symbols are related to single experiments and lines are the resulting means.

A) Population
B) Normalized

CASSIAR WET-MILLED FIBERS (Bulk)

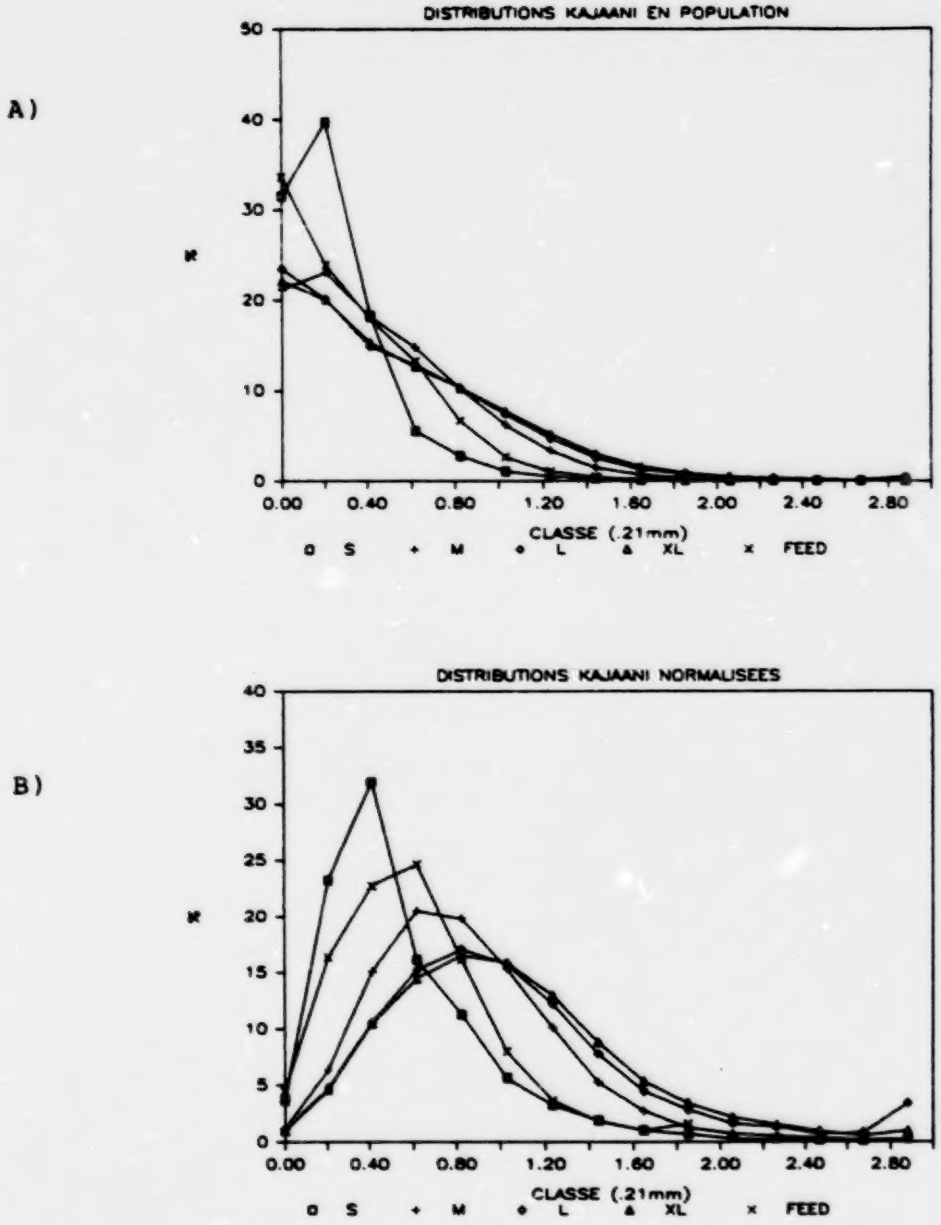


FIG. VIII.7a) Length distributions of wet-milled S, M. L, XL and FEED fibers obtained from a FS-100 KAJAANI Length Analyser, by .21 mm classes

Cassiar S:□ ; M: + ; L:◇ ; XL:△ ; FEED: X

A) Population
B) Normalized

(Class labels on X-coordinates give the lower limit of each class.)

WOODSREEF WET-MILLED 10-MESH FIBERS (Bulk)

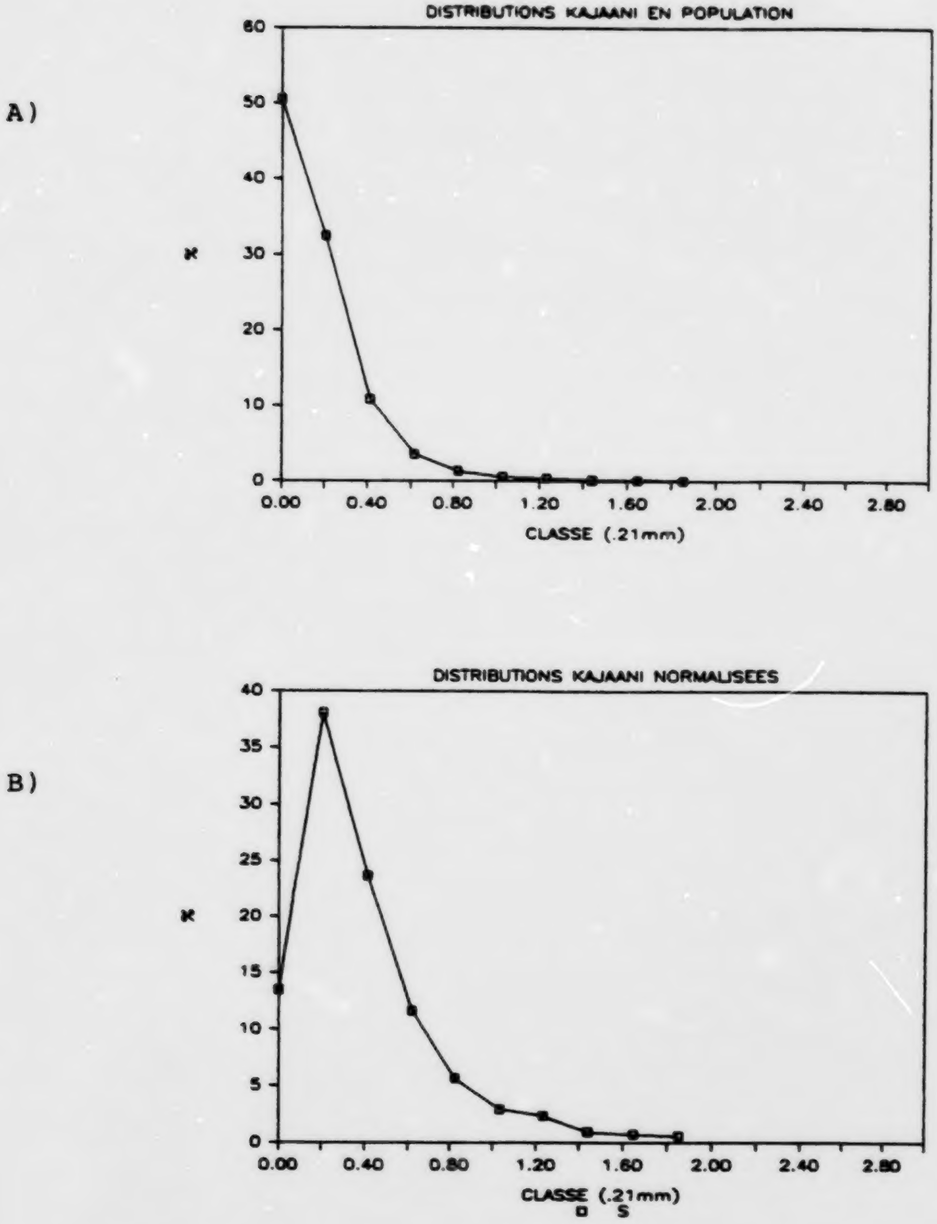


FIG. VIII.7b) Length distributions of wet-milled Woodsreef 10-mesh fibers obtained from a FS-100 KAJAANI Fiber Length Analyser, by .21 mm classes

A) Population
B) Normalized

CASSIAR DRY-MILLED FIBERS (Bulk)

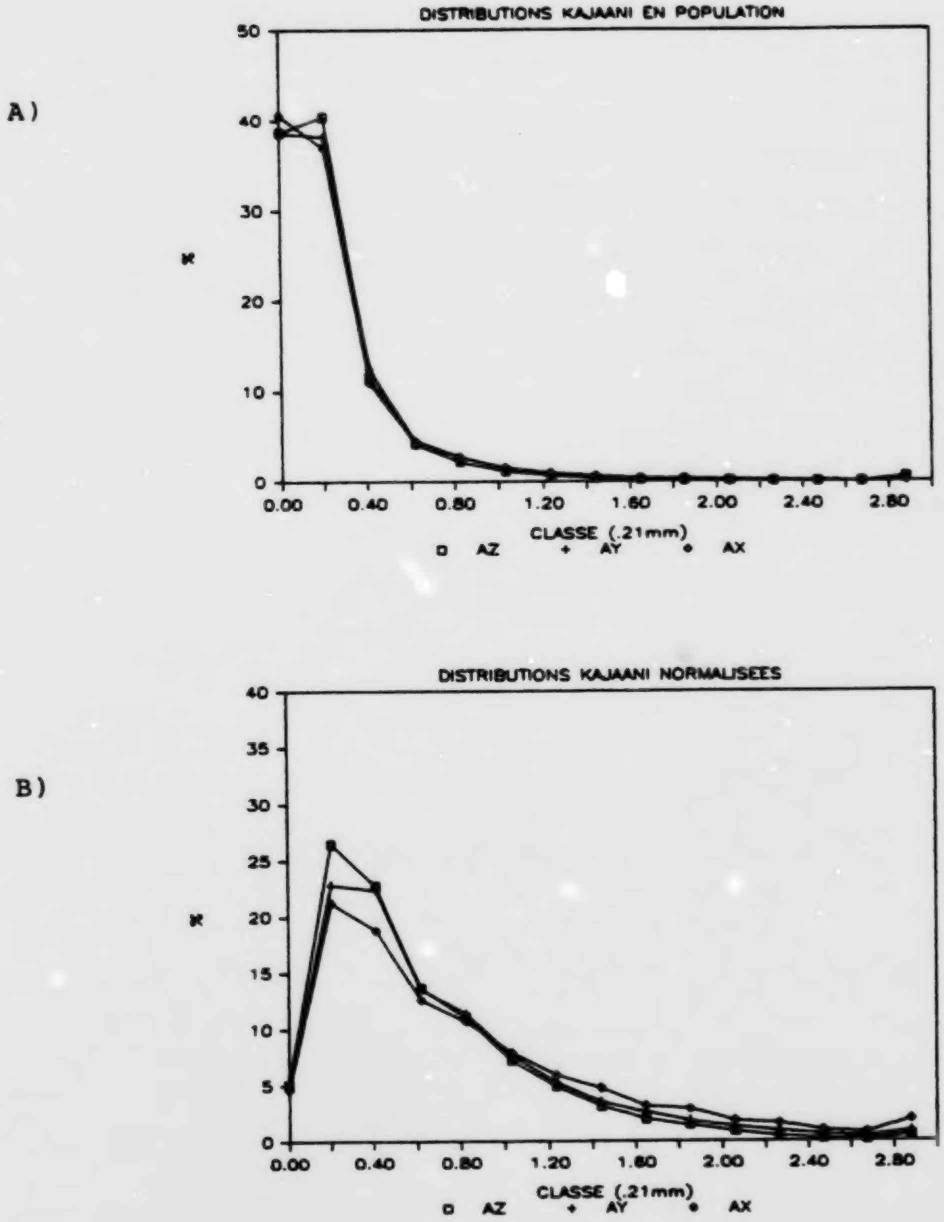


FIG. VIII.7c) Length distributions of wet-milled AZ, AY, AX fibers obtained from a FS-100 KAJAANI Fiber Length Analyser, by .21 mm classes

Cassiar AZ:□ ; AY: + ; AX:◇

b) Pallmann opened fibers

The Kajaani FS-100 Fiber Length Analyzer was also used to determine the length distributions of the various fiber products after Pallmann opening.[1] The distributions (population and normalized) obtained for the wet-milled Cassiar and Woodsreef fibers are grouped in Fig. VIII.8a, b; corresponding data for the dry-milled Cassiar fibers AZ, AY and AX are shown on Fig. VIII.8c. The computed mean lengths and standard deviations are given in Table VIII.6.

TABLE VIII.6: Mean lengths from Kajaani length distributions for various Pallmann (6500 rpm) opened fibers

Sample (Pallmann opened)	Population ℓ (mm)	Normalized $\bar{\ell}$ (mm)
Wet-milled		
Cassiar S	.193	.383
" M	.360	.583
" L	.403	.693
" XL	.383	.707
Woodsreef 10-mesh	.210	.423
Dry-milled		
Cassiar AZ	.217	.443
" AY	.230	.500
" AX	.243	.580

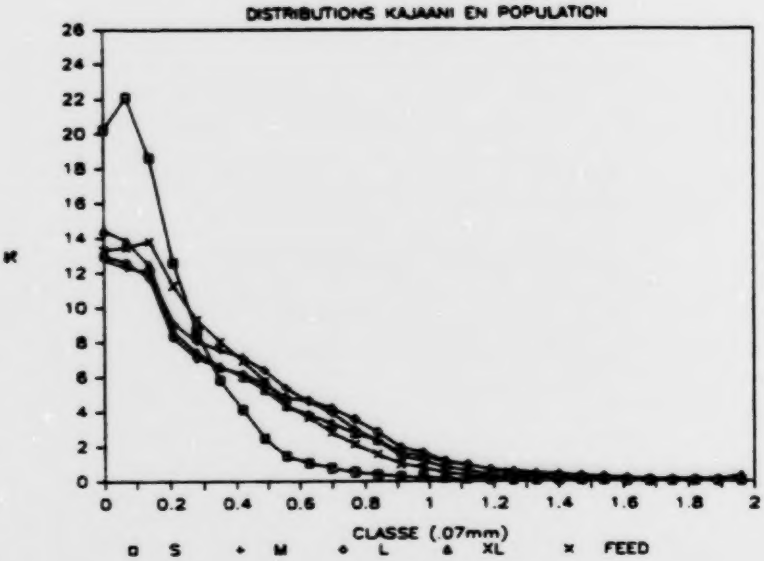
The mean length values computed by our Kajaani FS-100 instrument is incorrect; values are between 25 and 50% shorter than they should be based on the distributions.

---

[1] After Pallmann opening, the fibers were found to flow through the Kajaani capillary cell with only occasional clogging.

CASSIAR WET-MILLED FIBERS (Open)

A)



B)

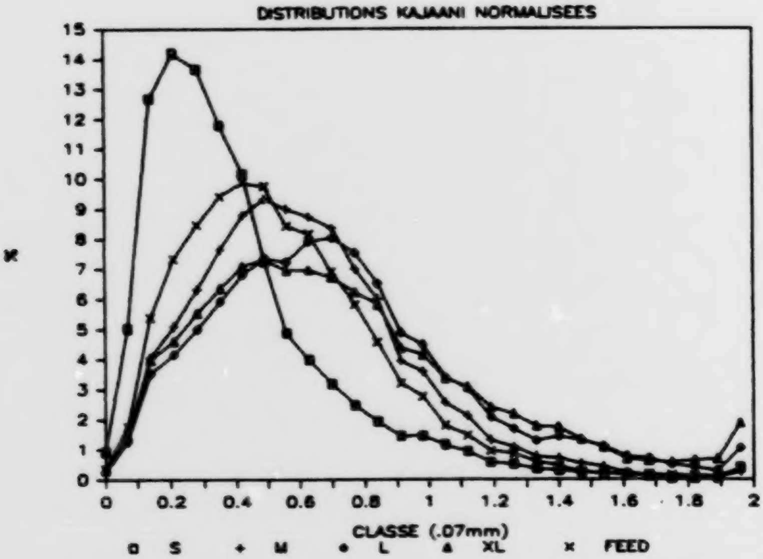


FIG. VIII.8a) Length distributions of wet-milled S, M, L, XL and FEED Pallmann opened fibers, obtained with a KAJAANI FS-100 Fiber Length Analyser, by .07 mm classes

Cassiar S: □ ; M: + ; L: ◇ ; XL: △ ; FEED: X

A) Population
B) Normalized

WOODSREED WET-MILLED 10-MESH FIBERS (Open)

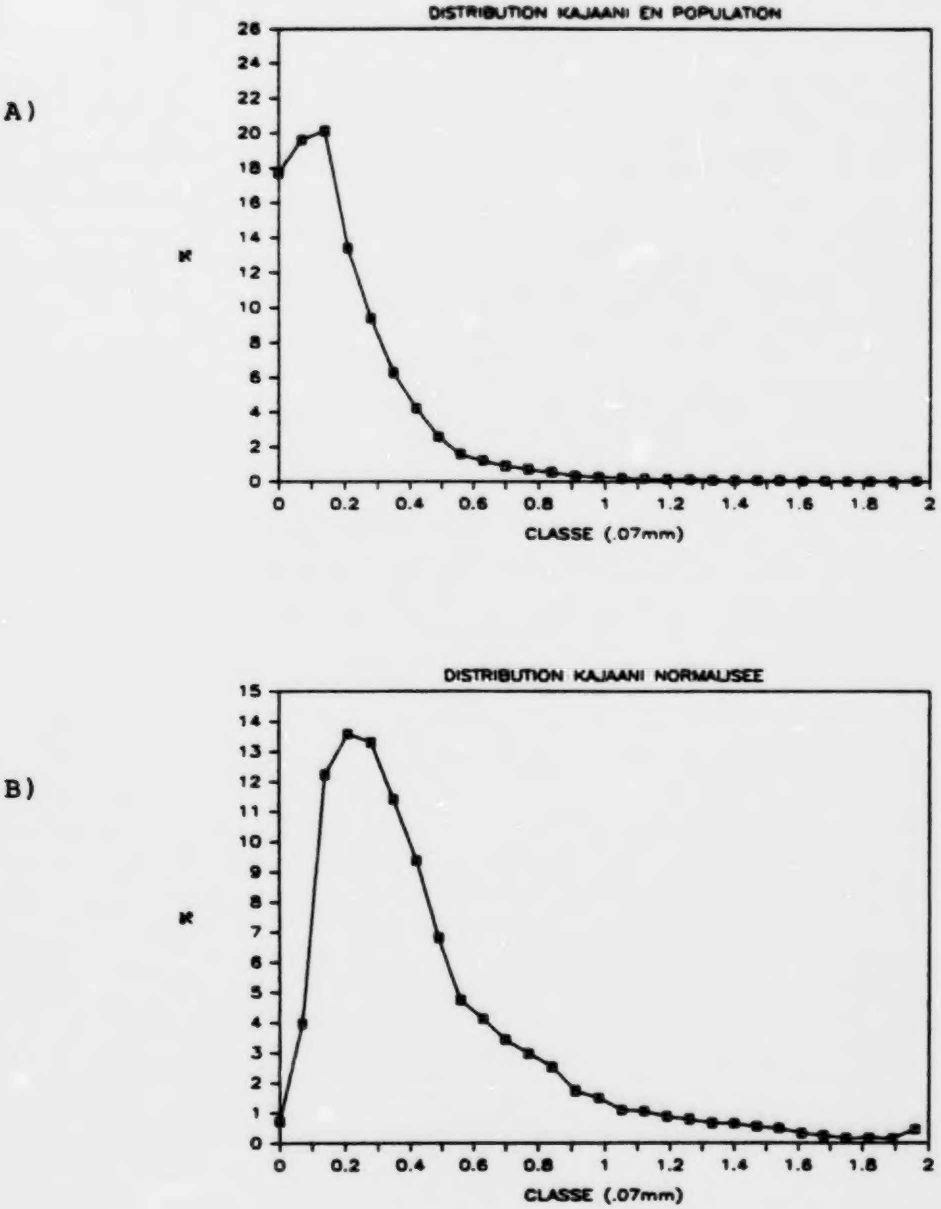


FIG. VIII.8b) Length distributions of wet-milled Woodsreef 10-mesh Pallmann opened fibers, obtained with a KAJAANI FS-100 Fiber Length Analyser, by .07 mm classes

A) Population
B) Normalized

CASSIAR DRY-MILLED FIBERS (Open)

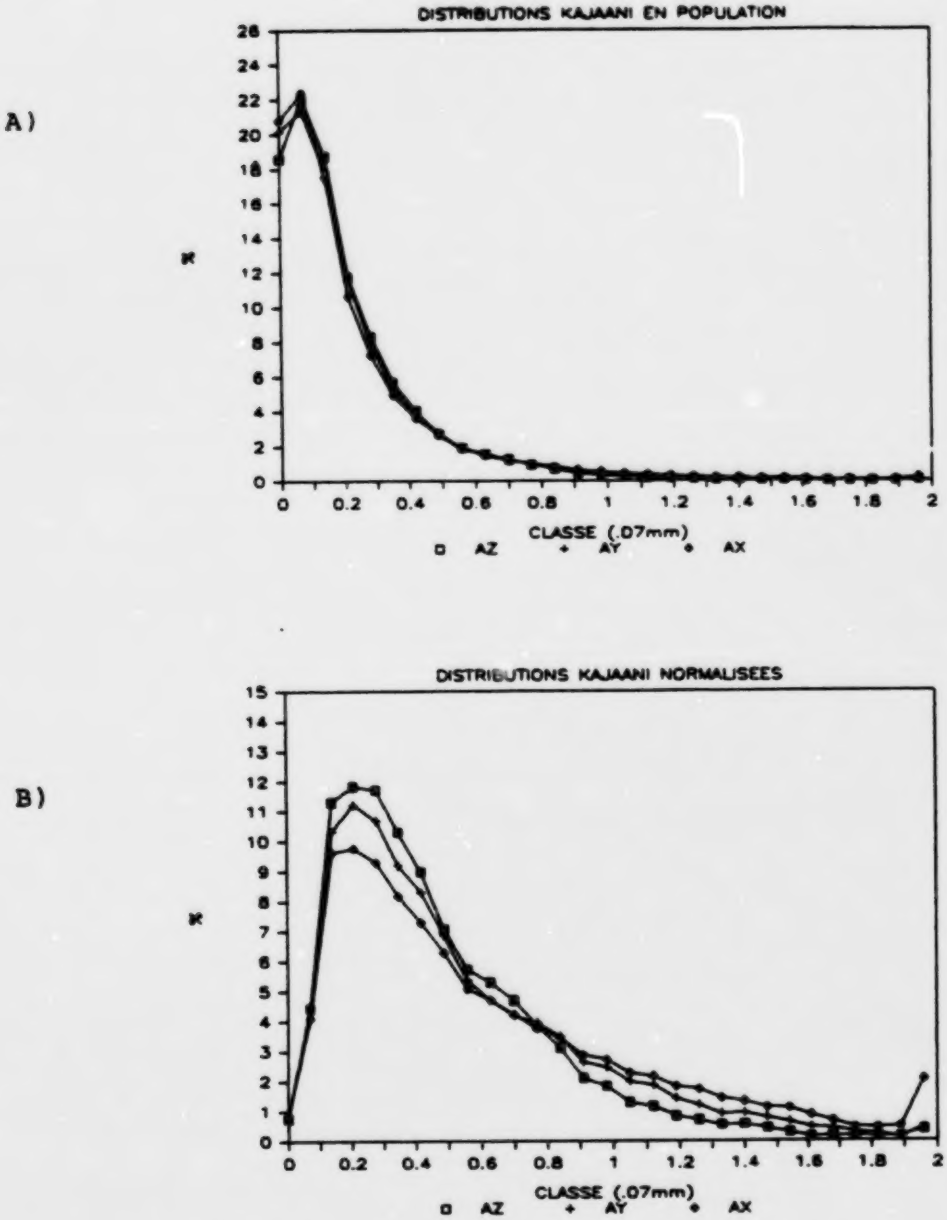


FIG. VIII.8c) Length distributions of dry-milled AZ, AY and AX Pallmann opened fibers, obtained with a KAJAANI FS-100 Fiber Length Analyser, by .07 mm classes

Cassiar AZ: □ ; AY: + ; AX: ◇

A) Population
B) Normalized

c) Kajaani FS-100 length distributions of wet-milled Cassiar M fibers opened by alternative wet processes

To confirm earlier observations regarding the relative effects of the Pallmann and other fiber opening procedures, we illustrate in Fig. VIII.9 the Kajaani length distributions of Cassiar wet-milled M fibers opened via different processes. The data shown compare the bulk M fibers and the same after Pallmann, wet turbine and Dounce tube opening.

Careful examination of the distributions, particularly the normalized distributions which emphasize the differences, reveals the following:

- compared to bulk (or normal) fibers, the Pallmann opened M fiber length distribution is shifted to lower length classes (as noted in Section VIII.3);
- the fiber length distributions of M fibers opened by either the wet turbine or the Dounce are not significantly different from the bulk M fibers.

The latter observation is clearly highly relevant for the development of fiber length measurement protocols.

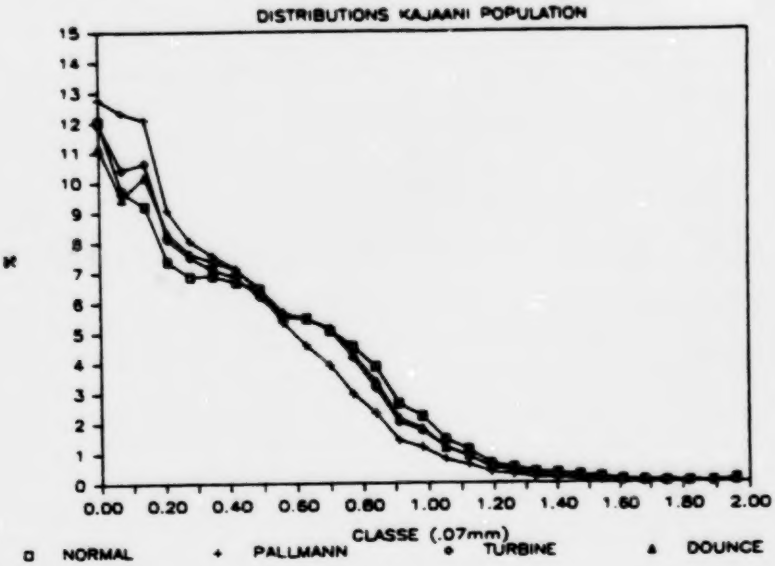
d) Preliminary conclusions on fiber length measurements using the Kajaani FS-100 analyser

The Kajaani fiber length data reported for various types of asbestos fibers (wet- and dry-milled, bulk and opened) support the following statements:

- Fiber length distributions can be obtained rapidly (5-10 minutes) with excellent statistical significance.
- Some coarse fibers, for example bulk wet-milled fibers (FEED) and bulk dry-milled fibers pose difficulties due to frequent clogging of the Kajaani capillary cell.

CASSIAR WET-MILLED M FIBERS

A)



B)

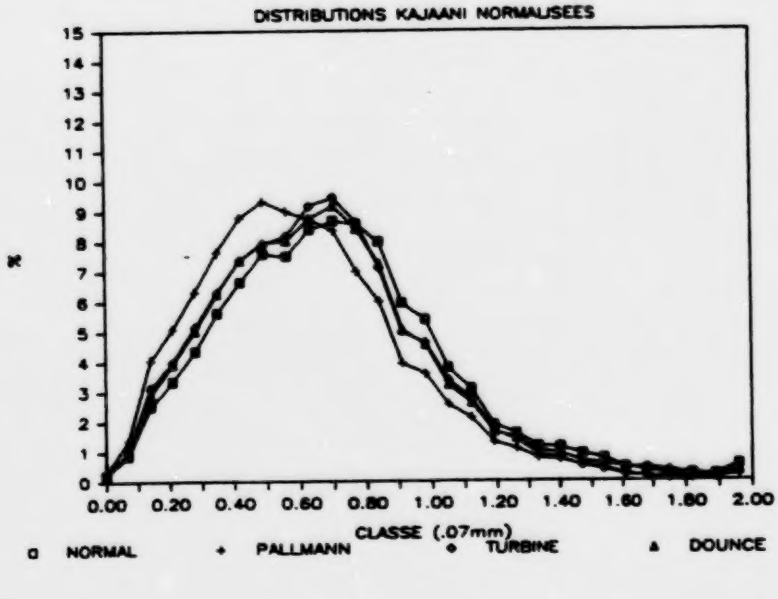


FIG. VIII.9) KAJAANI FS-100 length distributions of Cassiar wet-milled M fibers (bulk) and following Pallmann, turbine or Dounce opening

- The distributions obtained for subgroups of wet-milled fibers (S, M, L and XL), or dry-milled fibers (AZ, AY, AX) exhibit the expected qualitative order in distribution or mean length values.

- The mean lengths values computed by our Kajaani FS-100 instrument were incorrect, lying 25 to 50% lower than the expected values computed from the raw data. The length distributions are most certainly correct as they closely matched the optical microscopy data.

The above remarks indicate obvious qualitative advantages of the Kajaani Fiber Length Analyser for determination of fiber length distributions. Quantitative comparisons of the Kajaani data with optical microscopy results and with Bauer-McNett data will be discussed further in Section XI.2.

IX - RESULTS OF STANDARD CHRYSOTILE FIBER TESTS

In this section, we report the results of several standard tests, performed on Cassiar fibers, namely the Bauer-McNett wet classification and the Turner & Newall elutriation results; data on Wet Volumes, Freeness, T&N Magnetic Ratings and Rapid Surface Areas are also reported below. A separate section will be devoted to results of Fiber Strength Unit measurements.

IX.1 - Bauer-McNett wet classification

The results of Bauer-McNett wet classification analyses are given in Table IX.1 and illustrated as a bar chart in Fig. IX.1.

CASSIAR FIBERS

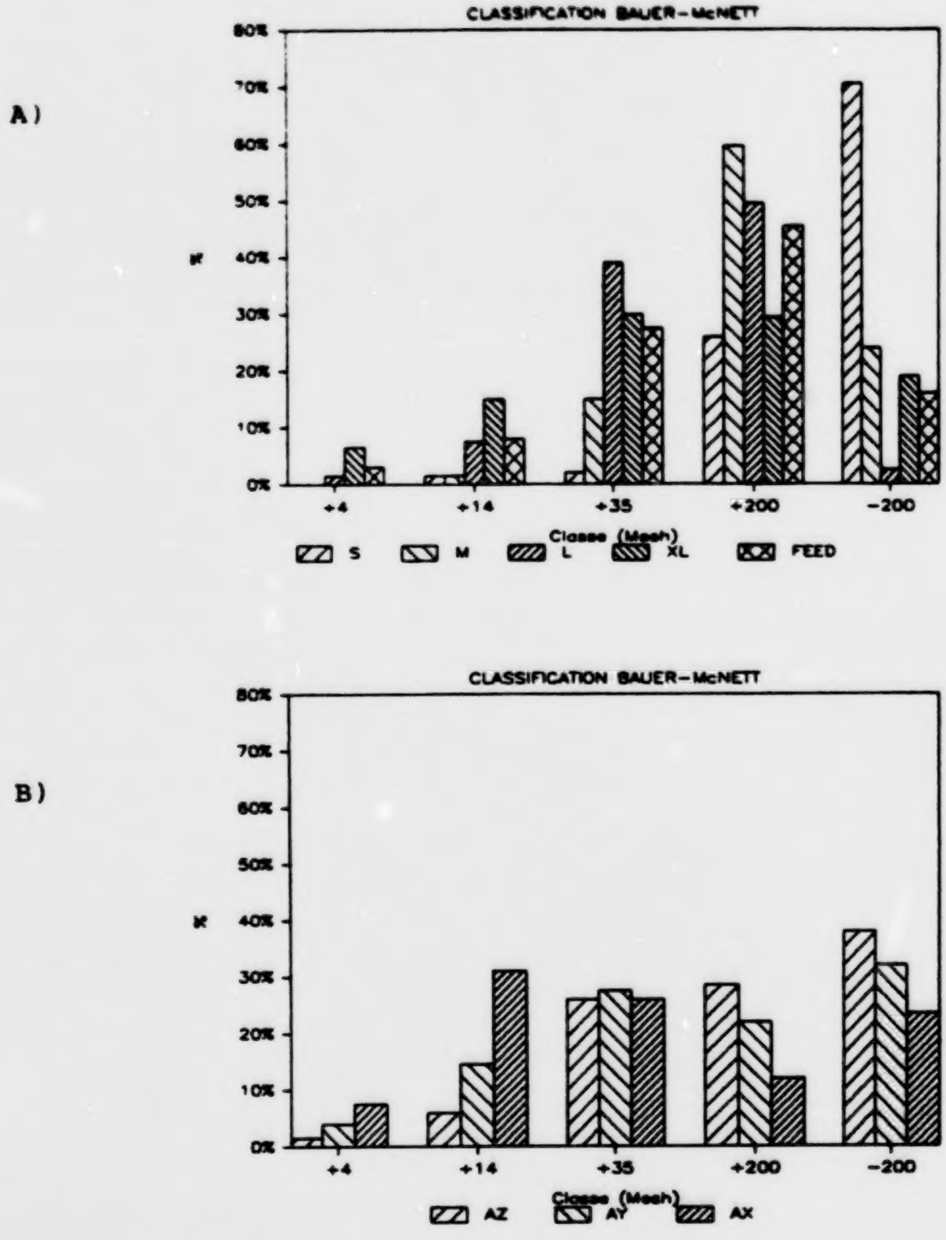


FIG. IX.1 Bar chart representation of the Bauer-Mcnett results for wet- and dry-milled Cassiar fibers

A) Wet-milled fibers
B) Dry-milled fibers

TABLE IX.1: Bauer-McNett classification of Cassiar fibers. (Classes in Tyler Mesh standard designation, values in brackets are apperture sizes in mm.)

Sample	+4 [4.75 mm]	+14 [1.18 mm]	+35 [.425 mm]	+200 [.075 mm]	-200 [.075 mm]
Wet-milled					
S	0	1.5	2	26	70.5
M	0	1.5	15	59.5	24
L	1.5	7.5	39	49.5	2.5
XL	6.5	15	30	29.5	19
FEED	3	8	27.5	45.5	16
Dry-milled					
AZ	1.5	6	26	28.5	38
AY	4	14.5	27.5	22	32
AX	7.5	31	26	12	23.5

As might be expected, the BMN data show higher contents of the +4 and +14-mesh fractions with the longer fiber types, i.e., comparing S, M, L, and XL fibers or AZ, AY and AX. It is also quite striking that the S fibers contain an exceptionally high -200-mesh fraction, with almost 97 wt % passing through 35-mesh (the latter corresponds to a .425 mm apperture size). By conventional interpretation, the S grade would be viewed as consisting largely of dust mixed with some proportion of very short fibers. The other wet-milled fibers, M to XL exhibit BMN fractions more evenly distributed, it is also noteworthy that the M fibers contain a very low -200-mesh fraction.

From the Bauer-McNett data for each of the classified wet-milled fibers (S, M, L, XL) and the relative weight of each grade in the FEED product, we have reconstructed the Bauer-McNett distribution for a hypothetical classified FEED. The experimental and calculated Bauer-McNett fractions are compared in Fig. IX.2a, and differences between these distributions are illustrated for each class in Fig. IX.2b. The comparison shows that the wet classification process used at Cassiar leads to a mild fiber opening; the latter is evidenced by slight decreases in the three longer fiber classes (+4, +14 and +35-mesh) and corresponding increases in the shortest classes (+200 and -200-mesh).

IX.2 - T&N elutriation

The results of T&N elutriation measurements performed on wet- and dry-milled Cassiar fibers are reported in Table IX.2.

TABLE IX.2: T&N elutriation results of bulk Cassiar fibers (in %)

Sample	Crude	Partial	Open	-200
Wet-milled				
S	.4	.8	44.8	54.0
M	3.6	2.6	78.0	15.8
L	10.55	5.65	83.85	0
XL	7.20	3.4	74.0	15.4
FEED	4.75	7.65	87.6	0
Dry-milled				
AZ	14.8	13.2	41.8	30.2
AY	22.8	14.0	38.0	25.2
AX	34.8	16.0	30.6	18.6

CASSIAR WET-MILLED FEED FIBERS

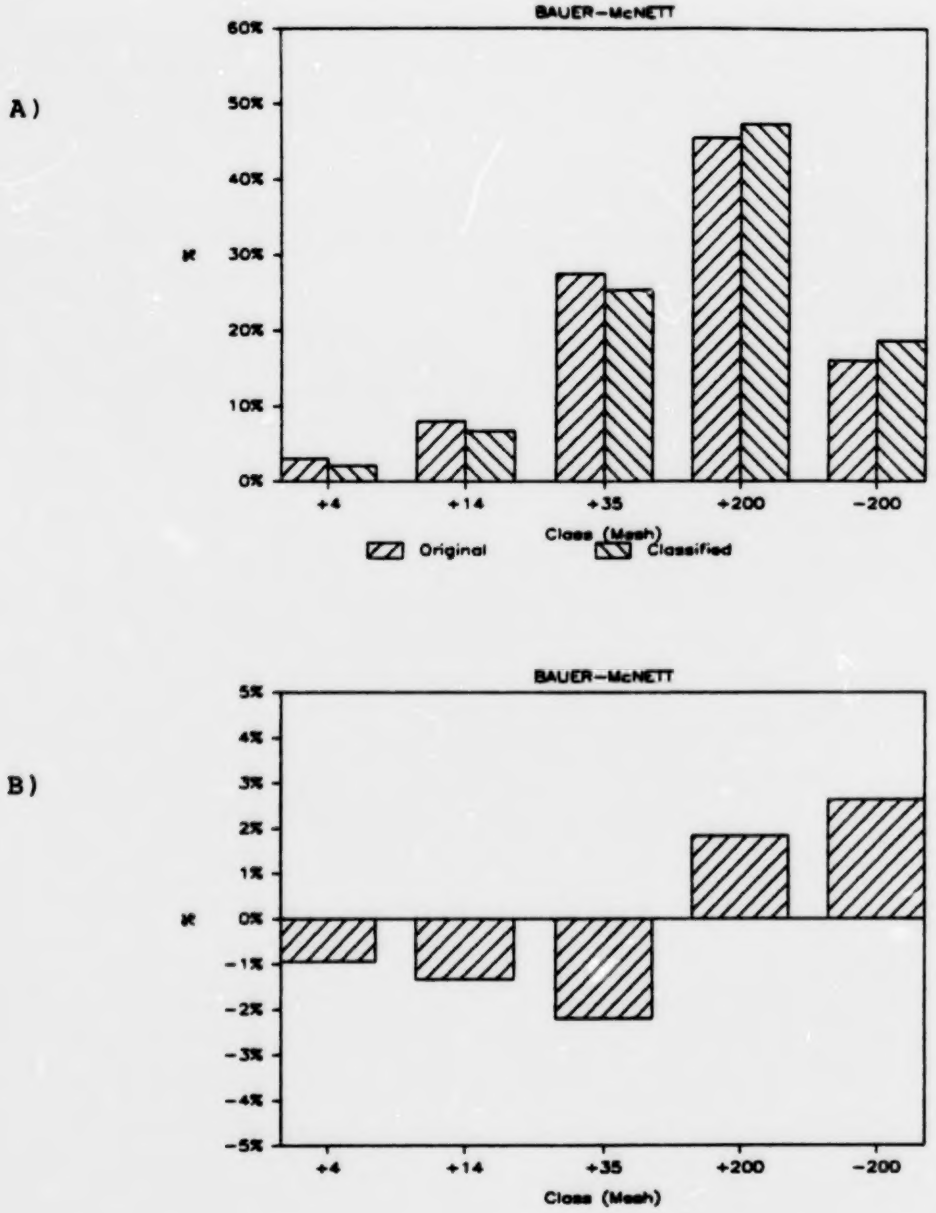


FIG. IX.2 A) Bauer-McNett classification results:
original: results obtained on the FEED material
classified : weighted sum of BMN fractions found in the wet-classified products (S, M, L, XL)

B) Differential bar chart: "classified" versus "original"

The T&N elutriation data follow a pattern similar to the Bauer-McNett classification data, the S fibers again appearing quite different from the others. This qualitative agreement may be expected in view of the similar physical principle exploited in these two methods. In later discussions, we will refer only to Bauer-McNett analysis.

IX.3 - Wet Volume, Freeness, T&N Magnetic Rating and Rapid Surface Area

The data on wet volume, freeness, T&N magnetic rating and rapid surface area obtained for Cassiar wet- and dry-milled fibers are collected in Table IX.3.

TABLE IX.3: Wet Volume, Freeness, T&N, Magnetic Rating and Surface Area results for bulk (normal) Cassiar fibers

Fiber type	Wet Volume (ml)	Freeness (sec)	T&N Mag. (MR)	RSA (dm^2/g)
Wet-milled				
S	1925	442	.56	>260*
M	1800	171	.28	190*
L	1875	163	.26	170*
XL	1825	126	.18	208*
FEED				238
Dry-milled				
AZ	400	113	3.36	100
AY	520	98	2.27	115
AX	520	45	1.59	86

* Carried out at Ceram-sna, Sherbrooke.

The wet volume values of wet-milled fibers are considerably higher than values normally found for dry-milled fibers. In fact, the wet-milled fibers fill almost completely the 2000 cm^3 water container used for this standard test. In accordance with these observations, the freeness results are high for the M, L and XL fibers, and very high for the S fibers. These general observations are normally associated with extensively opened chrysotile fibers.

The magnetic rating values of wet-milled fibers are found to be very low, indicating low magnetite contents. This observation is confirmed by the low levels of iron oxides found in these fibers from elemental analysis (Section VI.1). The S fibers shows a relatively high magnetic rating probably due to the concentration of fine magnetite particles in this fiber group.

X - RESULTS OF ASBESTOS-CEMENT STRENGTH TEST

Standard Fiber Strength Unit (FSU) tests were performed on the various Cassiar wet- and dry-milled fiber products and the results are collected in Table X.1; related data such as the weight fraction of fiber used in the cement mix, the adjusted modulus of rupture (MRA) and the loose density of the fibers after the opening step (one pass through a Pallmann mill at 6500 or a turbine). For each fiber sample, ten asbestos-cement samples were prepared and tested; the results shown are the mean from all trials (detailed results are given in Appendix H). Specific aspects of the methodologies and results are discussed below.

TABLE X.1: Results of fiber strength tests (FSU)[1] for various fiber types

Fiber types	Opening Technique	Qty of fiber (%)	MRA[2] (kg/cm^2)	FSU	Loose Density (lb/pi^3)
Wet-milled S	Pallmann	4.3	186.7	---	0.7
S	"	6.0	188.7	---	
M	"	6.8	251.7	135.4	1.3
L	"	7.6	262.8	126.2	1.5
XL	"	6.8	256.7	138.0	1.3
FEED	"	7.5	275.0	133.3	
Wet-milled S	Turbine	6.2	202.6	---	
M	"	7.4	278.5	136.7	
L	"	7.9	254.4	117.8	
XL	"	7.2	274.9	138.8	
Wet-milled L:					
Micro-wave dried	Pallmann	7.6	289.8	138.1	1.5
Vacuum dried	"	7.8	268.2	125.3	1.6
Dry-milled AZ	Pallmann	16.3	255.9	57.8*	4.4
AZ	Ball mill/BOP	12.8	292.4	82.8*	---
AY	Pallmann	11.4	261.7	84.0	2.7
AX	"	8.8	256.4	106.6	1.9

1 - FSU tests performed at Ceram-sna, Sherbrooke.

2 - MRA: Adjusted modulus of rupture.

Unless specified otherwise, the wet-milled fibers which were opened through a Pallmann mill had been dried, first in a micro-wave oven (80-90% of water removed) and second, in a vacuum oven at 110°C.

* It is readily apparent that a single Pallmann opening step does not yield optimal fiber strength with Cassiar dry-milled products.

X.1 - FSU results for wet-milled Cassiar M, L, XL and FEED fibers

The M, L, XL wet-milled grades and the FEED fibers all performed very well in the FSU test, giving high FSU values compared to conventional dry-milled asbestos. These results are shown in Fig. X.1 and labelled "Pallmann".

The initial proportion of fibers used in the A/C mix were chosen based upon the bulk density values measured after Pallmann opening. FSU values predicted from bulk densities (i.e. through an empirical relation), initially suggested an excellent cement reinforcement behavior for the wet-milled products. Preliminary rupture tests allowed final adjustment of fiber contents for the extensive test, and the rupture modulii (MRA) obtained were indeed near the values required by the test specifications (256-282 kg/cm^2). Thus, with respect to the fiber strength testing protocols, the wet-milled fibers (M, L, XL, FEED), after drying and Pallmann opening behave qualitatively like clean dry-milled fibers.

The drying procedure used to obtain a fiber suitable for Pallmann opening comprised first a micro-wave heating step to evaporate 80-90% of the water initially present and, second, vacuum oven heating at 110°C for 24 hrs. The possible effect of the fiber drying method on fiber strength was verified by submitting a sample of L fibers to extended microwave drying, and a similar sample to vacuum oven drying only. As the results in Table X.1 show, the microwave dried sample exhibit an appreciable increase in FSU rating, whereas no significant difference is seen for the vacuum oven dried sample.

The most plausible interpretation of the improved performance of the sample submitted to extensive microwave drying, is that some of the structural water involved fiber-fiber binding was removed, therefore facilitating further opening. This would seem worthy of future investigation.

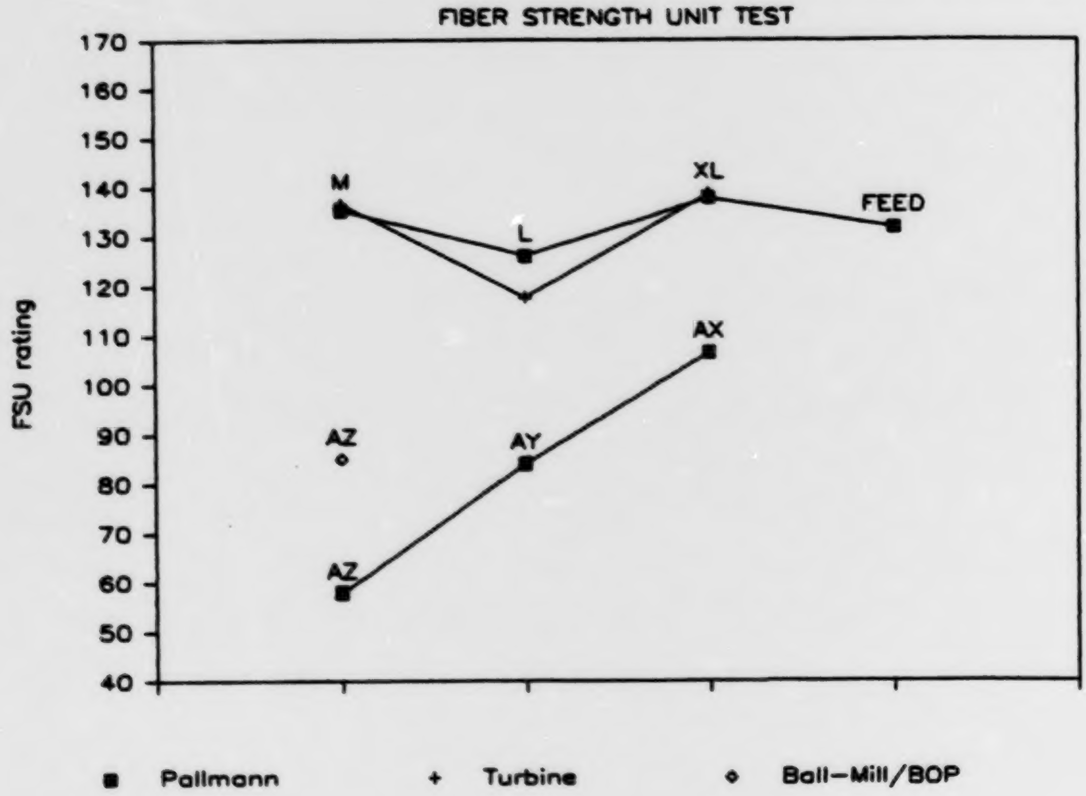


FIG. X.1 Fiber strength tests results of the wet- and dry-milled Cassiar fibers

(■) Pallmann opened
(+) Turbine opened
(◇) Ball-mill/BOP opened

X.2 - FSU results for wet-milled Cassiar S fibers

The unusually low bulk density of the S fibers after Pallmann opening lead to a very high predicted FSU ratings for these fibers (i.e., calculated from the empirical correlation). A first test carried out with a fiber content of 4,3% yielded a good, although too low MRA value. The results of a second test at a 6.0% fiber content showed only a marginal increase in the MRA value. It must therefore be concluded that the S fiber group cannot achieve the required MRA valued for A/C products, although it provides good cement reinforcement at low fiber contents.

X.3 - FSU results for turbine opened wet-milled fibers (S, M, L, XL, FEED)

A turbine opening device was tested as a possible wet opening method for FSU measurements or for later inclusion in a wet-milling circuit. As judged by the wet volumes observed, the extent of fiber opening achieved with the turbine for all grades (S, M, L, XL, FEED) was very similar to that obtained through Pallmann milling.

The results of fiber strength test obtained with turbine-opened fibers are very consistent with those obtained from the Pallmann-opened fibers. A comparison of FSU values for Pallmann- and turbine-opened fibers (Fig. X.1) show that the only significant discrepancy found was for the L grade, an effect for which we have not confirmed presently. The low MRA values found earlier with the S fibers (not shown) were confirmed in this set of measurements.

XI - COMPARISONS AMONG METHODS FOR FIBER LENGTH MEASUREMENT OR CLASSIFICATION

This section provides detailed comparisons and relative assessments of the various methods exploited here for the measurement of fiber lengths, namely:

- optical microscopy
- Kajaani Fiber Length Analyser
- Bauer-McNett classification BMN.

The primary aims of the analysis which follows, and one of the key concern of the present study, is the quantitative assessment of the Kajaani Fiber Length Analyser as a potential replacement for BMN tests.

XI.1 - Comparison of optical microscopy and Kajaani length distribution data

A qualitative comparison of length distributions obtained from optical microscopy (OM) and with the Kajaani FS-100 Analyser is illustrated for the various bulk fibers in Fig. XI.1(a-i). A similar set of distribution curves for the Pallmann opened fibers are shown in Fig. XI.2(a-i). In these comparisons the length classes used in OM measurements were matched to those of the Kajaani data. The computed mean lengths ($\bar{\ell}$) for all fiber types (bulk and opened) are collected in Table XI.1 for both the population and the length normalized distributions (we recall here that the mean values computed by our Kajaani FS-100 instrument were too low by 25-50%, but it could be shown that the problem lies only in the treatment of the data, not in the data themselves).

CASSIAR WET-MILLED S FIBERS (Bulk)

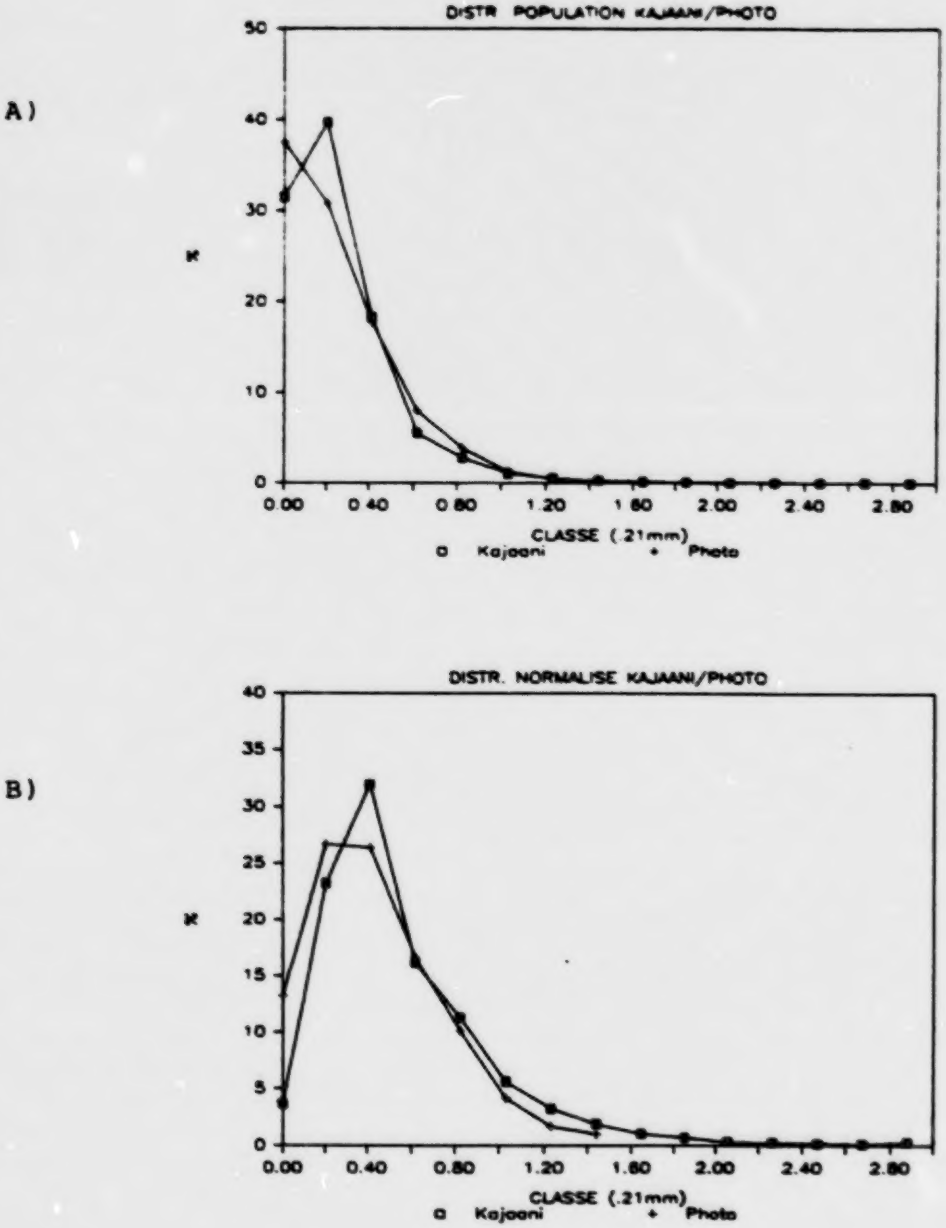


FIG. XI.1a) Comparison between KAJAANI (□) and optical microscopy (+) population length distributions of wet-milled S fibers, by .21 mm classes

A) Population
B) Normalized

(Class labels on X-coordinates give the lower limit of each class.)

CASSIAR WET-MILLED M FIBERS (Bulk)

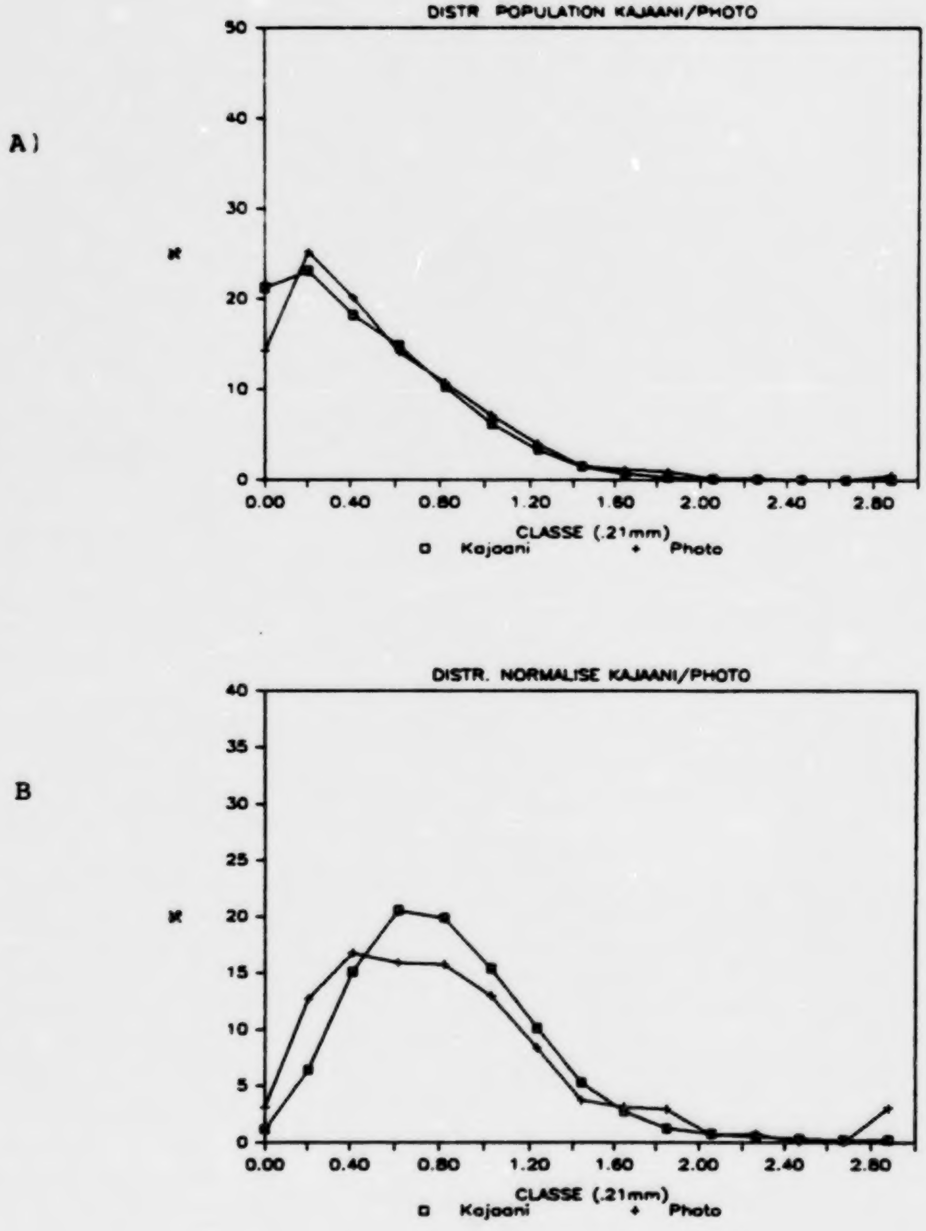


FIG. XI.1b) Comparison between KAJAANI (□) and optical microscopy (+) population length distributions of wet-milled M fibers, by .21 mm classes

CASSIAR WET-MILLED L FIBERS (Bulk)

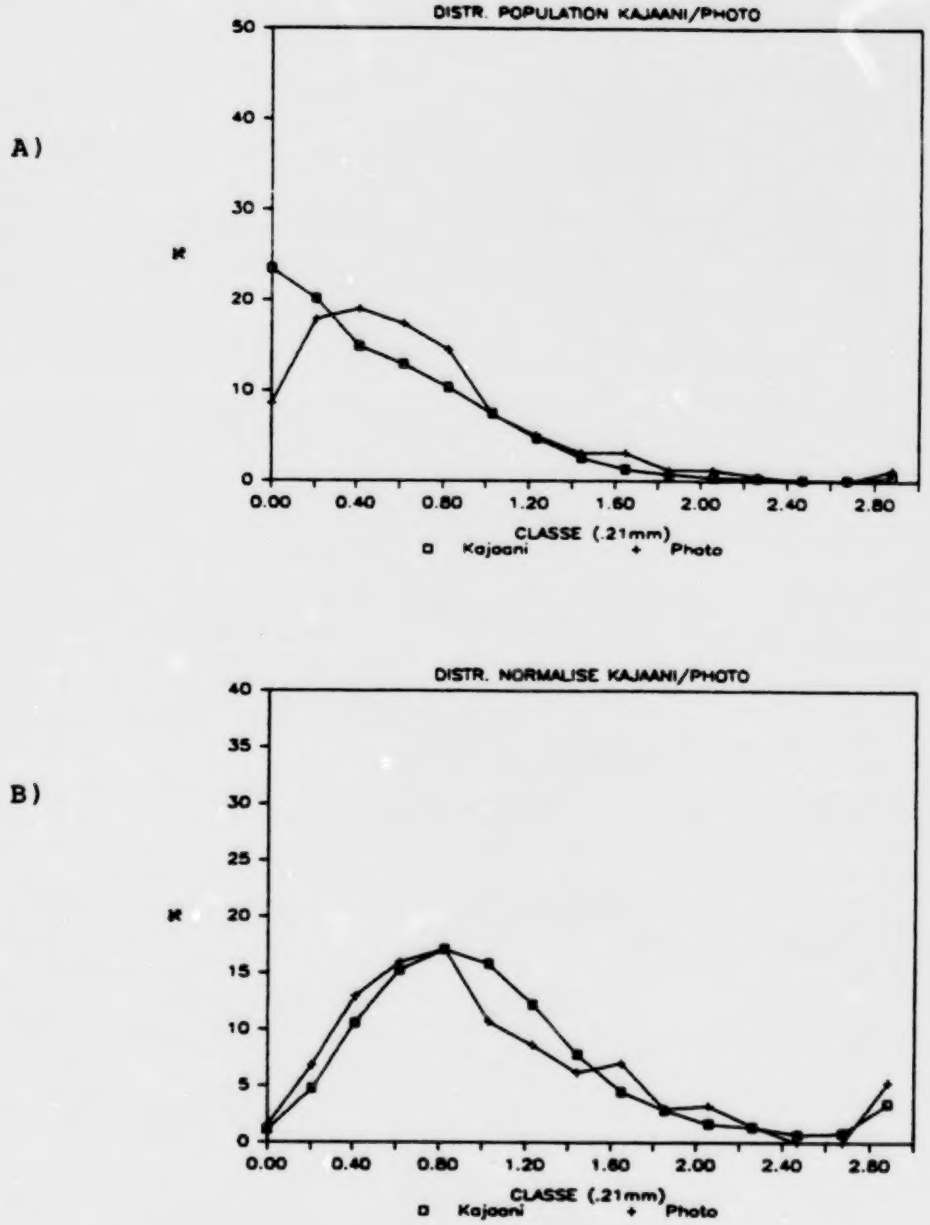


FIG. XI.1c) Comparison between KAJAANI (□) and optical microscopy (+) population length distributions of wet-milled L fibers, by .21 mm classes

CASSIAR WET-MILLED XL FIBERS (Bulk)

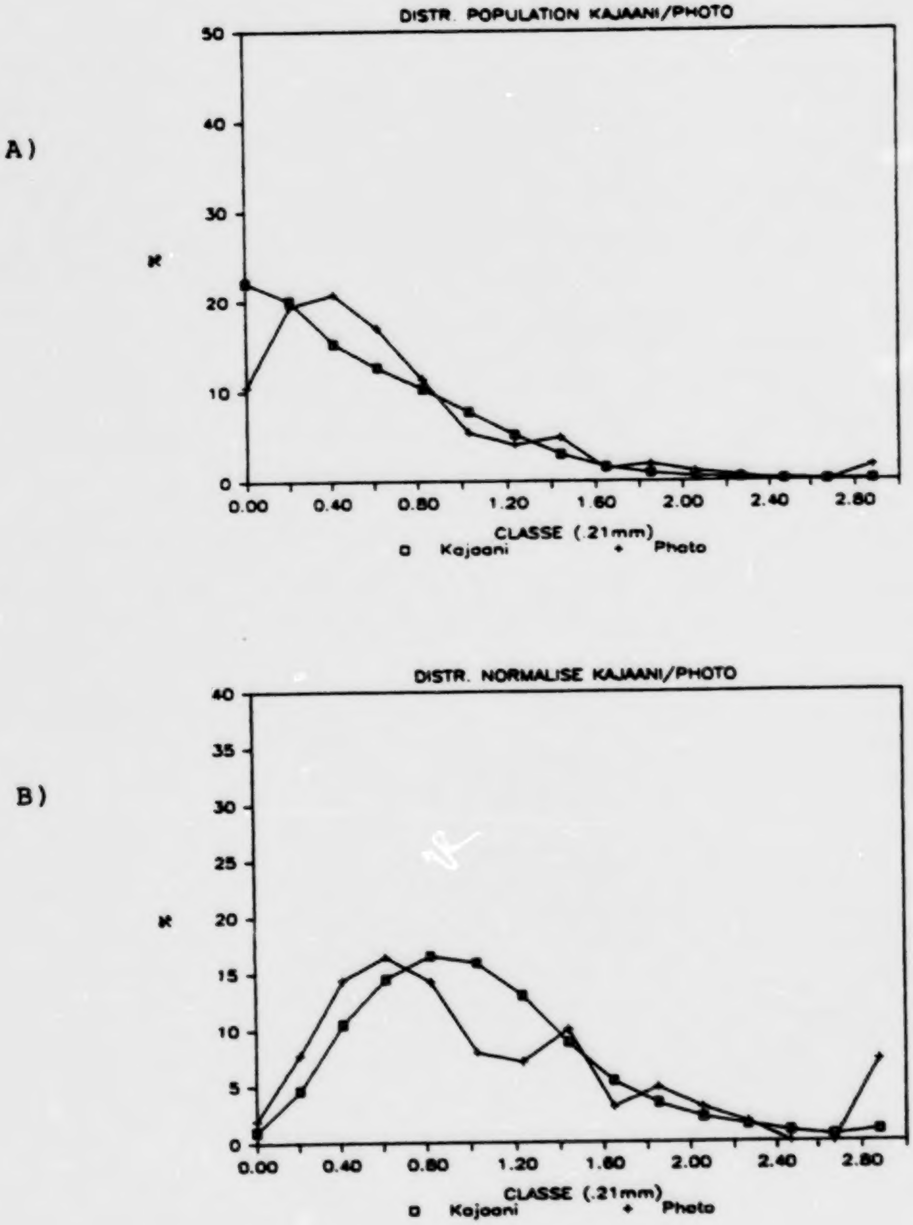


FIG. XI.1d) Comparison between KAJAANI (□) and optical microscopy (+) population length distributions of wet-milled XL fibers, by .21 mm classes

CASSIAR WET-MILLED FEED FIBERS (Bulk)

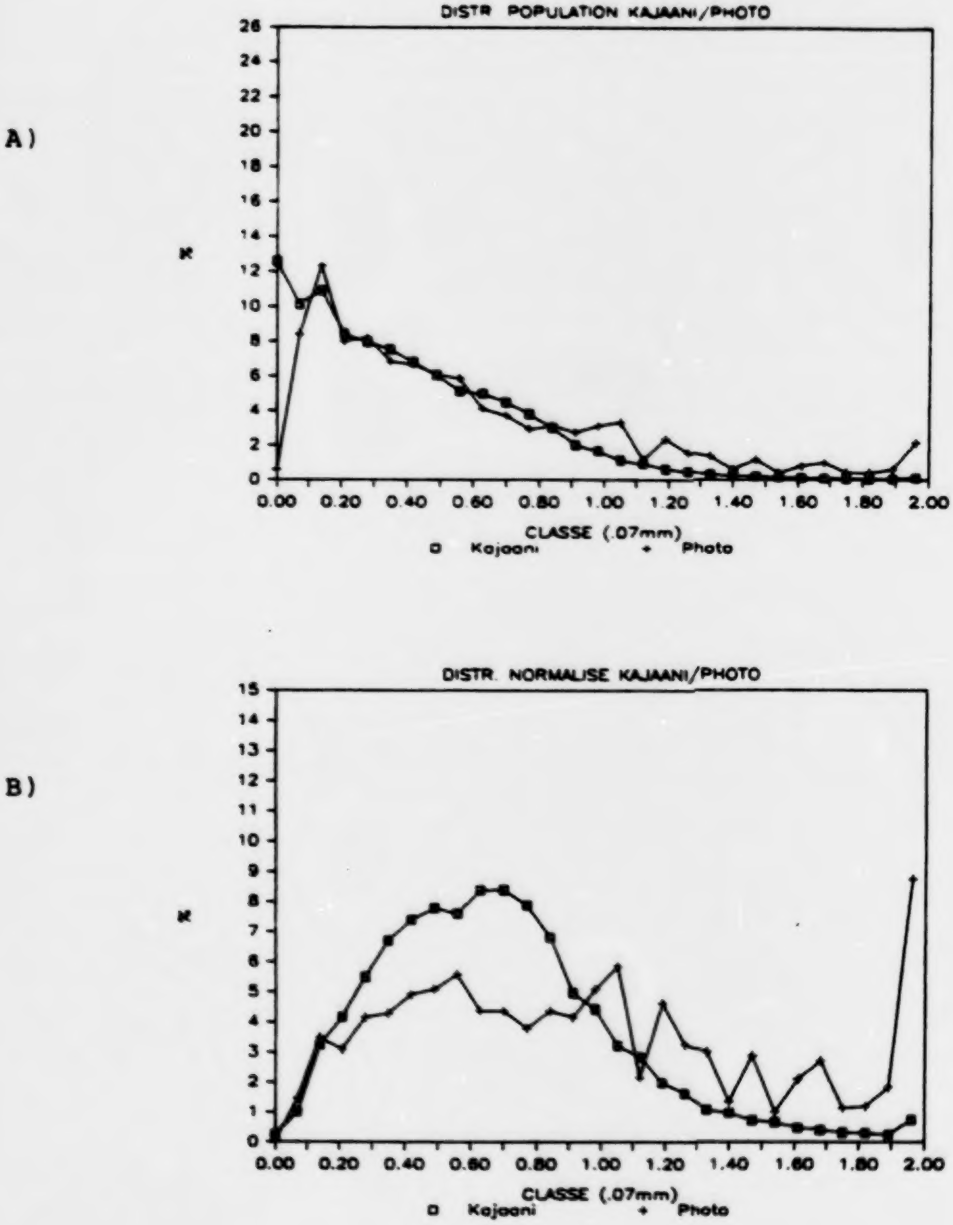


FIG. XI.1e) Comparison between KAJAANI (□) and optical microscopy (+) population length distributions of wet-milled FEED fibers, by .21 mm classes

WOODSREEF WET-MILLED 10-MESH FIBERS (Bulk)

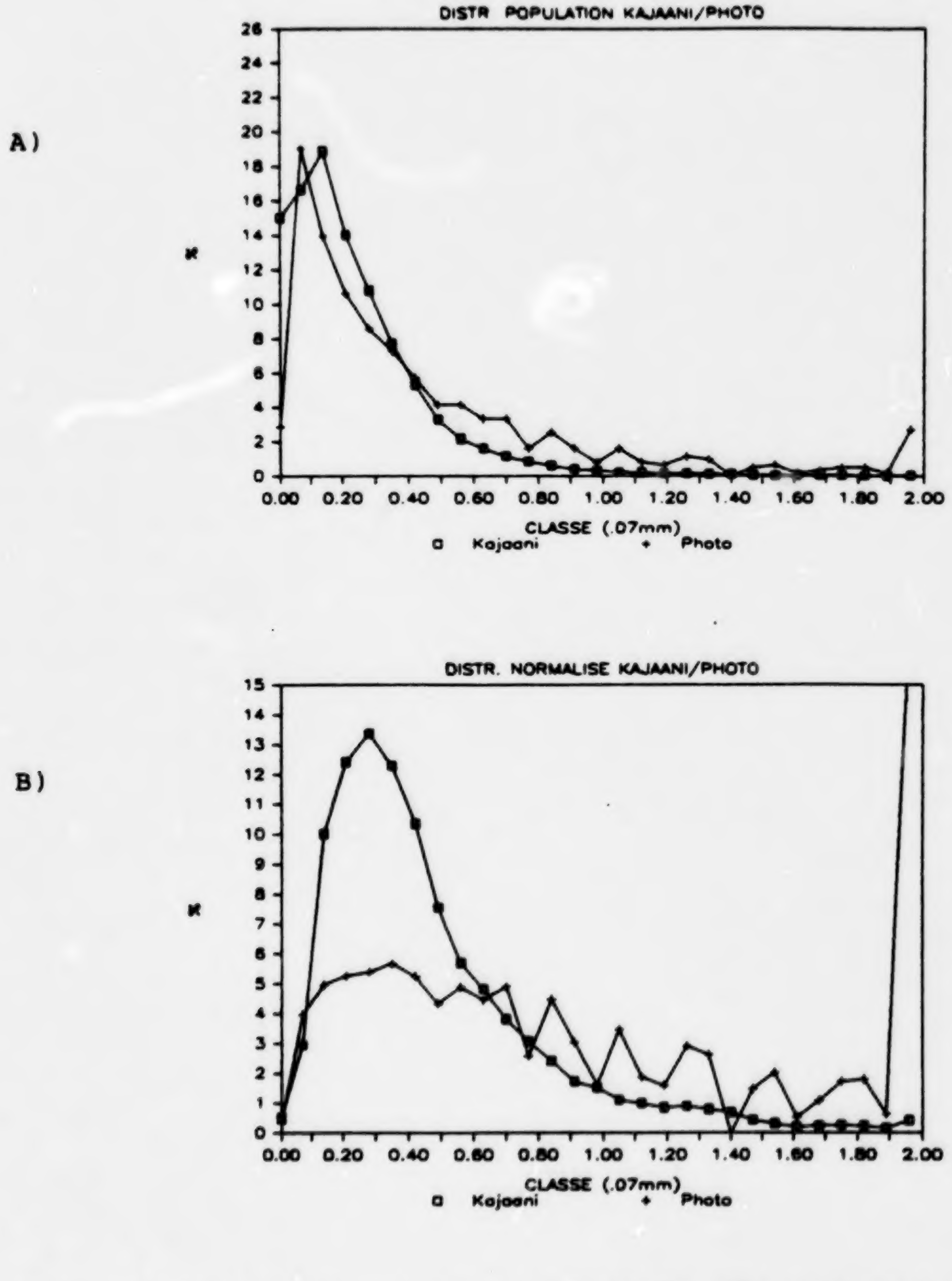
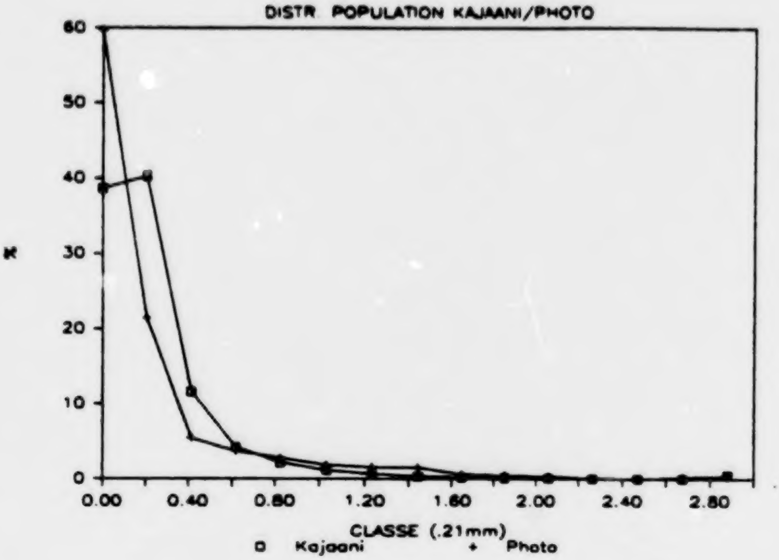


FIG. XI.1f) Comparison between KAJAANI (□) and optical microscopy (+) population length distributions of Woodsreef 10-mesh wet-milled fibers, by .21 mm classes

CASSIAR DRY-MILLED AZ FIBERS (Bulk)

A)



B)

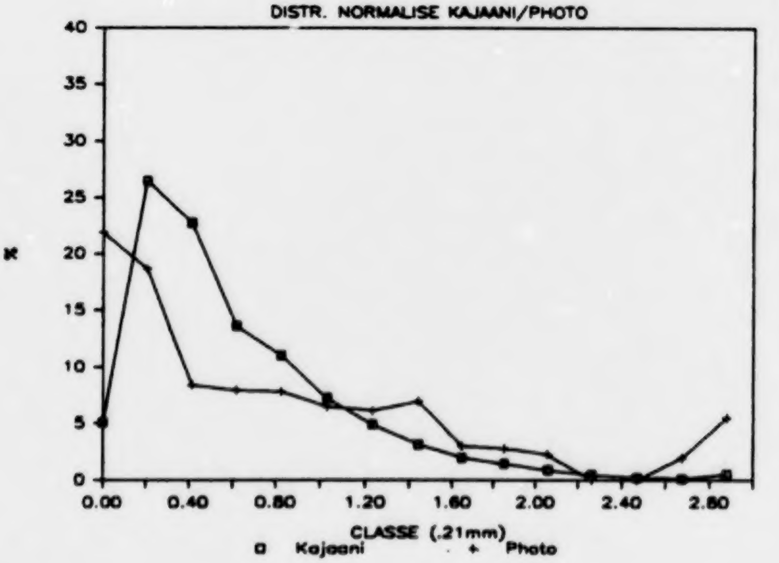


FIG. XI.1g) Comparison between KAJAANI (□) and optical microscopy (+) population length distributions of AZ dry-milled fibers, by .21 mm classes

CASSIAR DRY-MILLED AY FIBERS (Bulk)

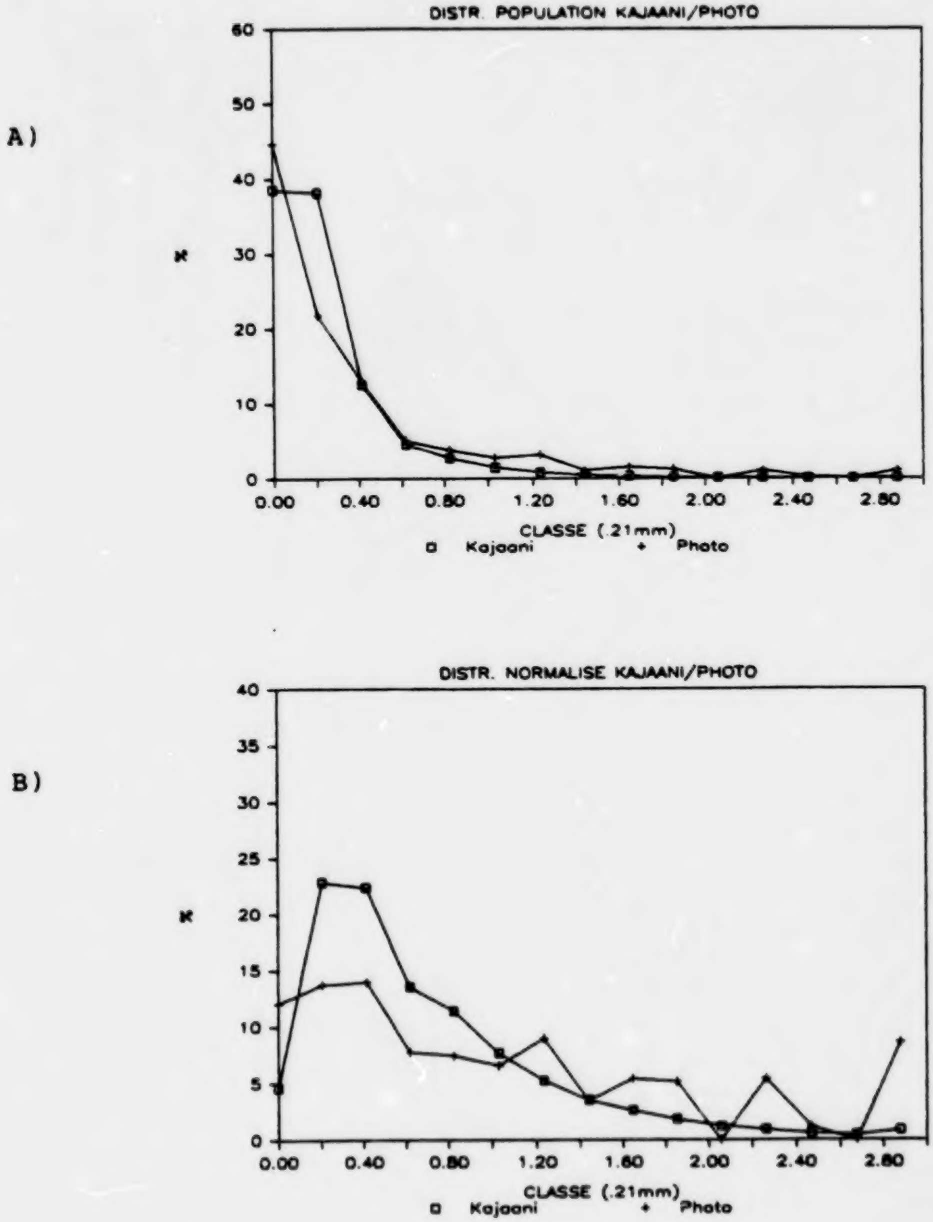


FIG. Xi.1h) Comparison between KAJAANI (□) and optical microscopy (+) population length distributions of AY dry-milled fibers, by .21 mm classes

CASSIAR DRY-MILLED AX FIBERS (Bulk)

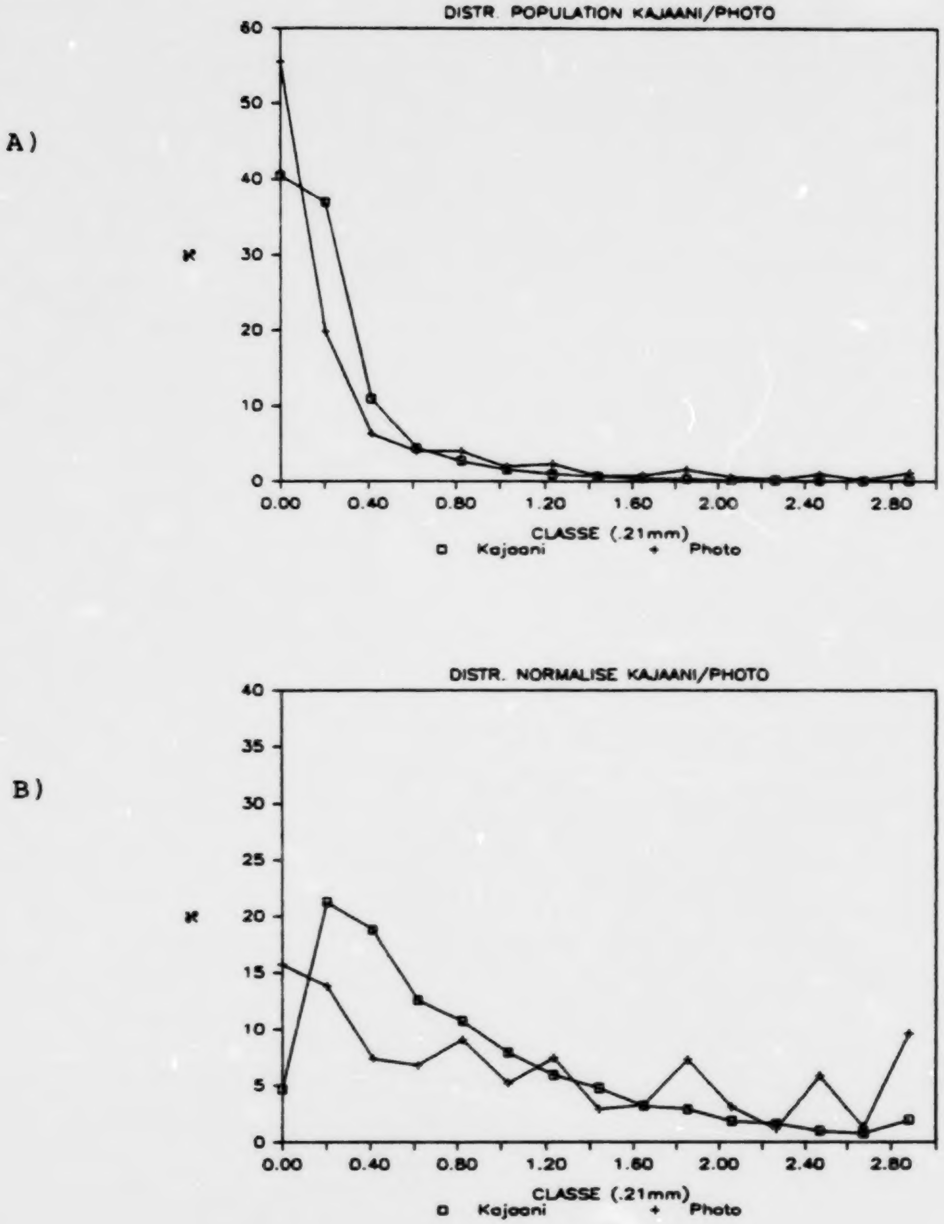


FIG. XI.1i) Comparison between KAJAANI (□) and optical microscopy (+) population length distributions of AX dry-milled fibers, by .21 mm classes

CASSIAR WET-MILLED S FIBERS (Open)

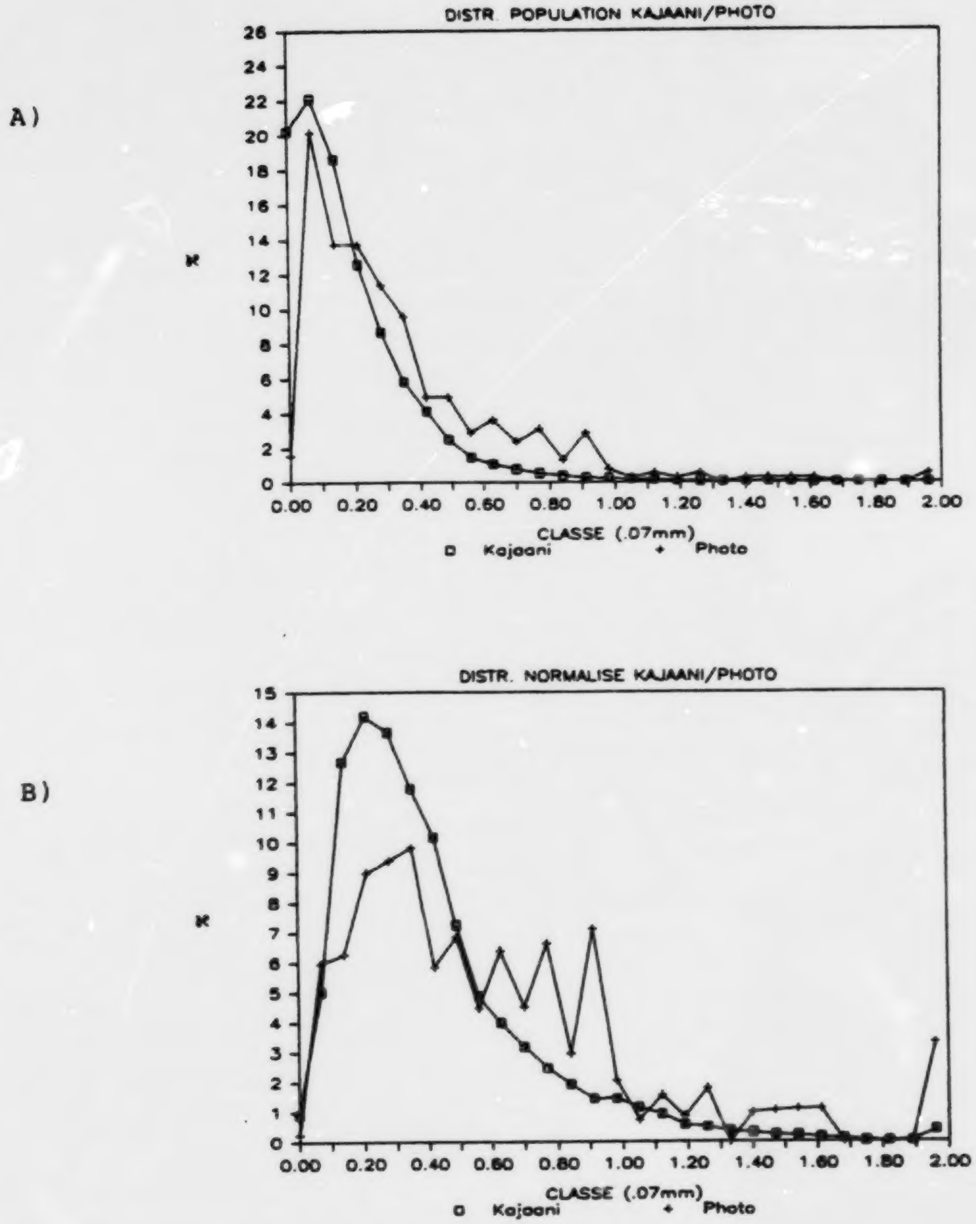


FIG. XI.2a) Comparison between KAJAANI (□) and optical microscopy (+) population length distributions of wet-milled S Pallmann opened fibers, by .21 mm classes

(Class labels on X-coordinates give the lower limit of each class)

CASSIAR WET-MILLED M FIBERS (Open)

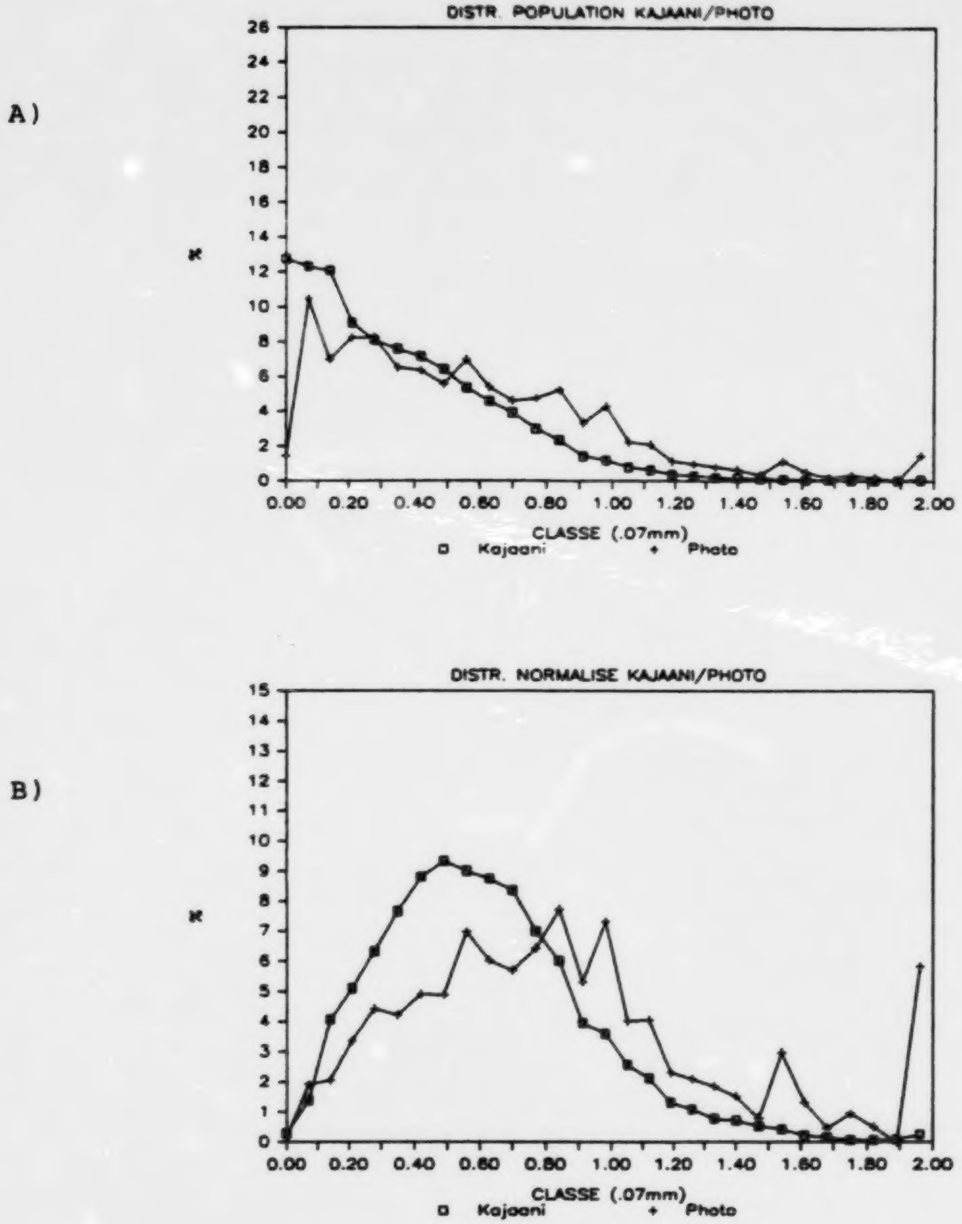
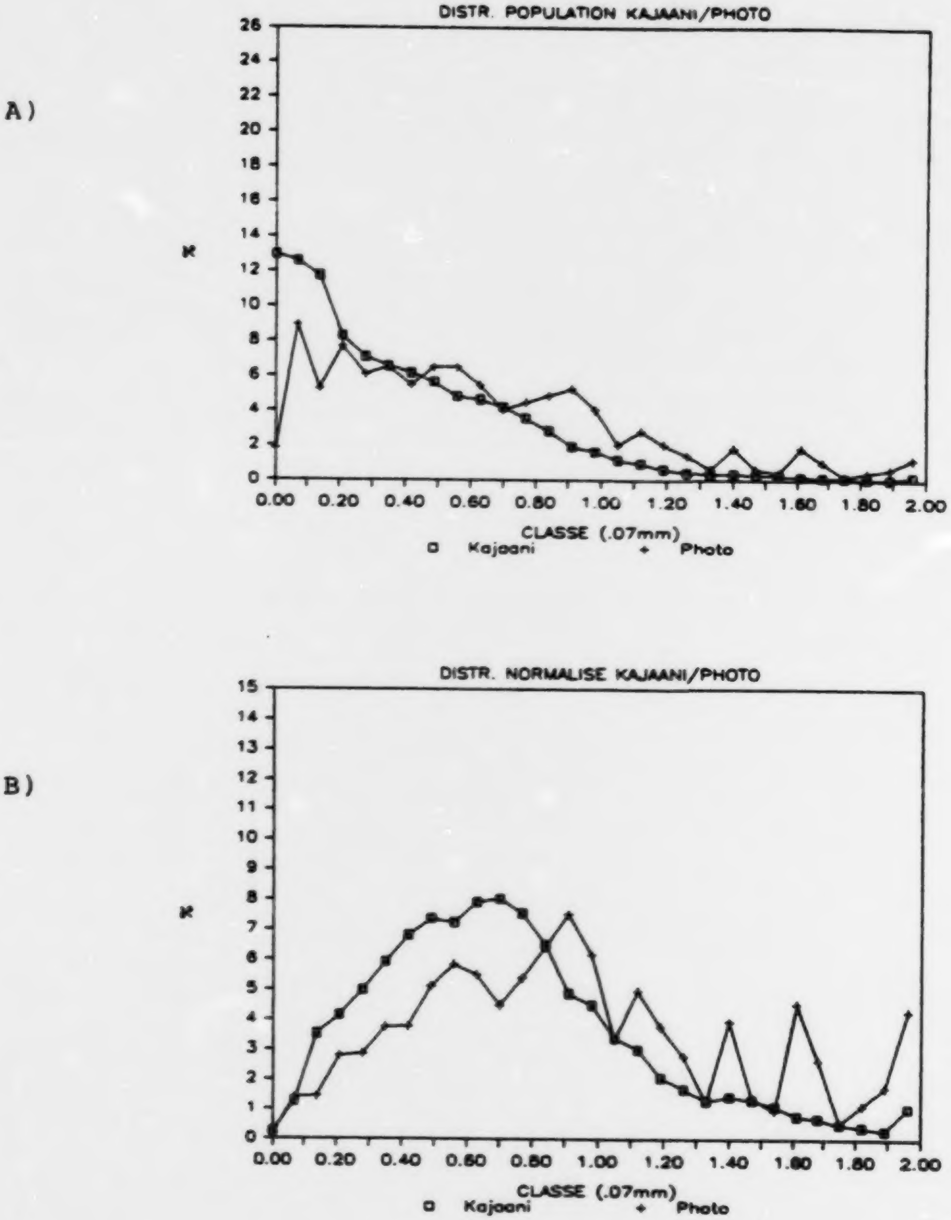


FIG. XI.2b) Comparison between KAJAANI (□) and optical microscopy (+) population length distributions of wet-milled M Pallmann opened fibers, by .21 mm classes

CASSIAR WET-MILLED L FIBERS (Open)



DISTR. POPULATION KAJAANI/PHOTO

DISTR. NORMALISE KAJAANI/PHOTO

FIG. XI.2c) Comparison between KAJAANI (□) and optical microscopy (+) population length distributions of wet-milled L Pallmann opened fibers, by .21 mm classes

CASSIAR WET-MILLED XL FIBERS (Open)

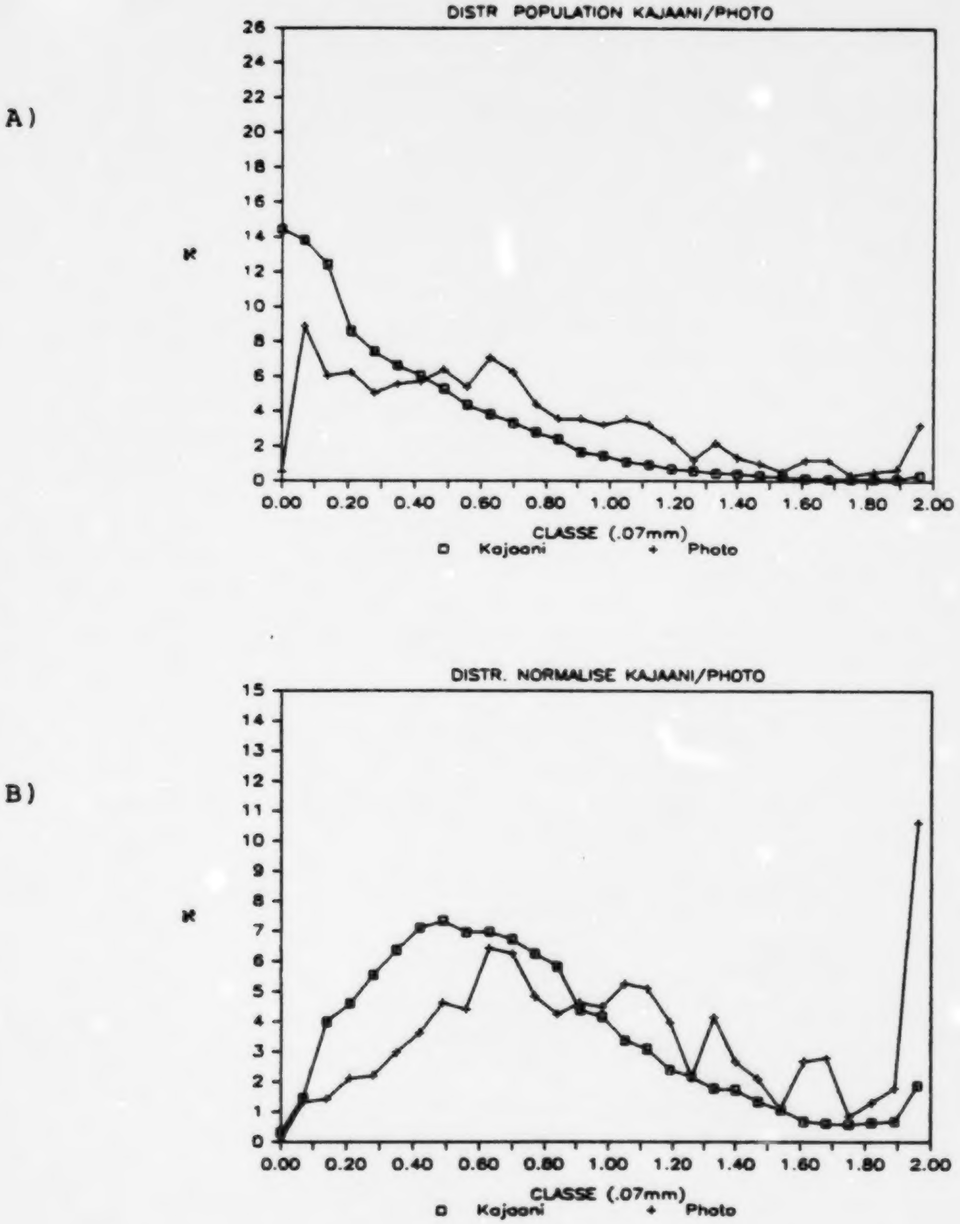


FIG. XI.2d) Comparison between KAJAANI (□) and optical microscopy (+) population length distributions of wet-milled XL Pallmann opened fibers, by .21 mm classes

CASSIAR WET-MILLED FEED FIBERS (Open)

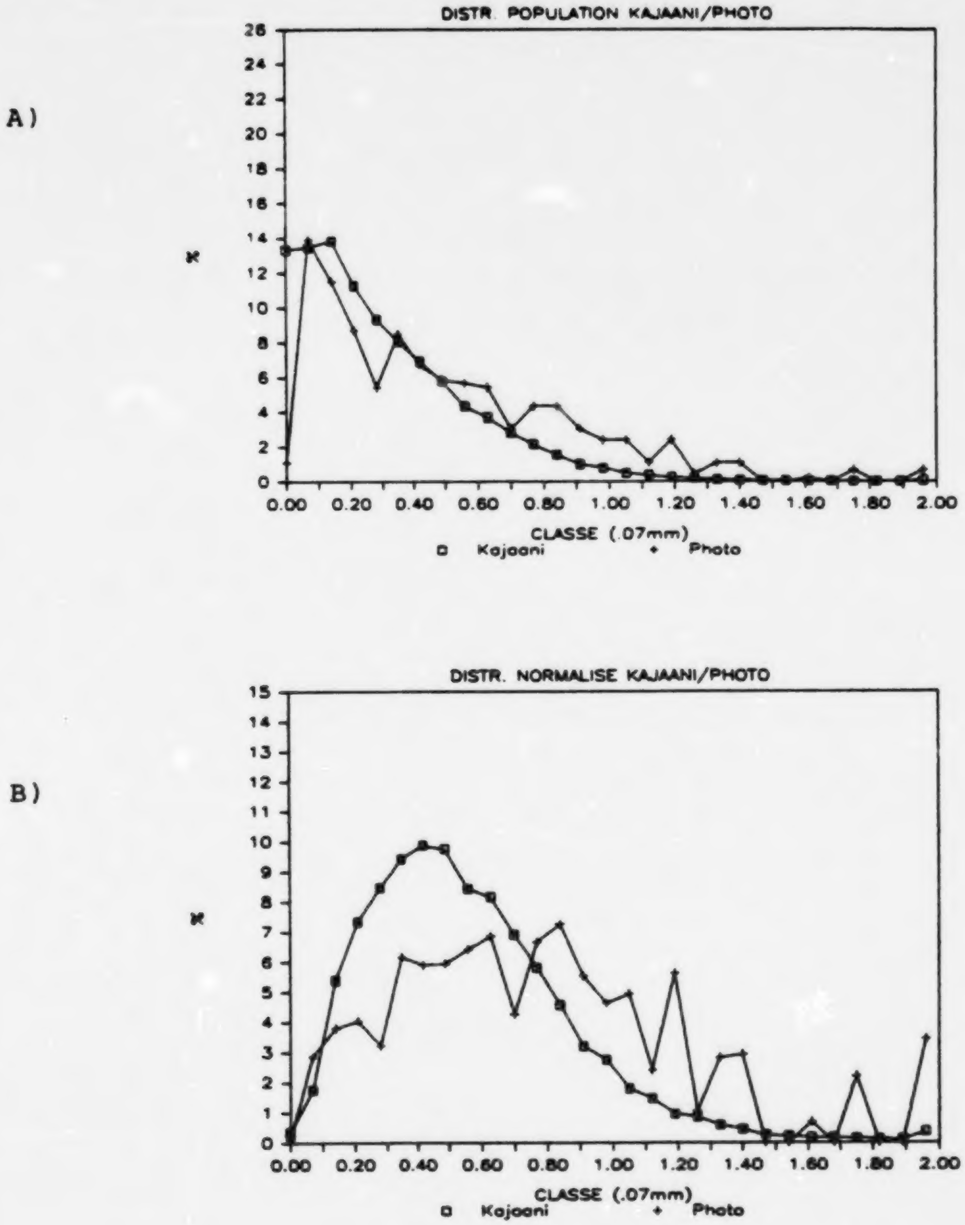


FIG. XI.2e) Comparison between KAJAANI (□) and optical microscopy (+) population length distributions of wet-milled FEED Pallmann opened fibers, by .21 mm classes

WOODSREEF WET-MILLED 10-MESH FIBERS (Open)

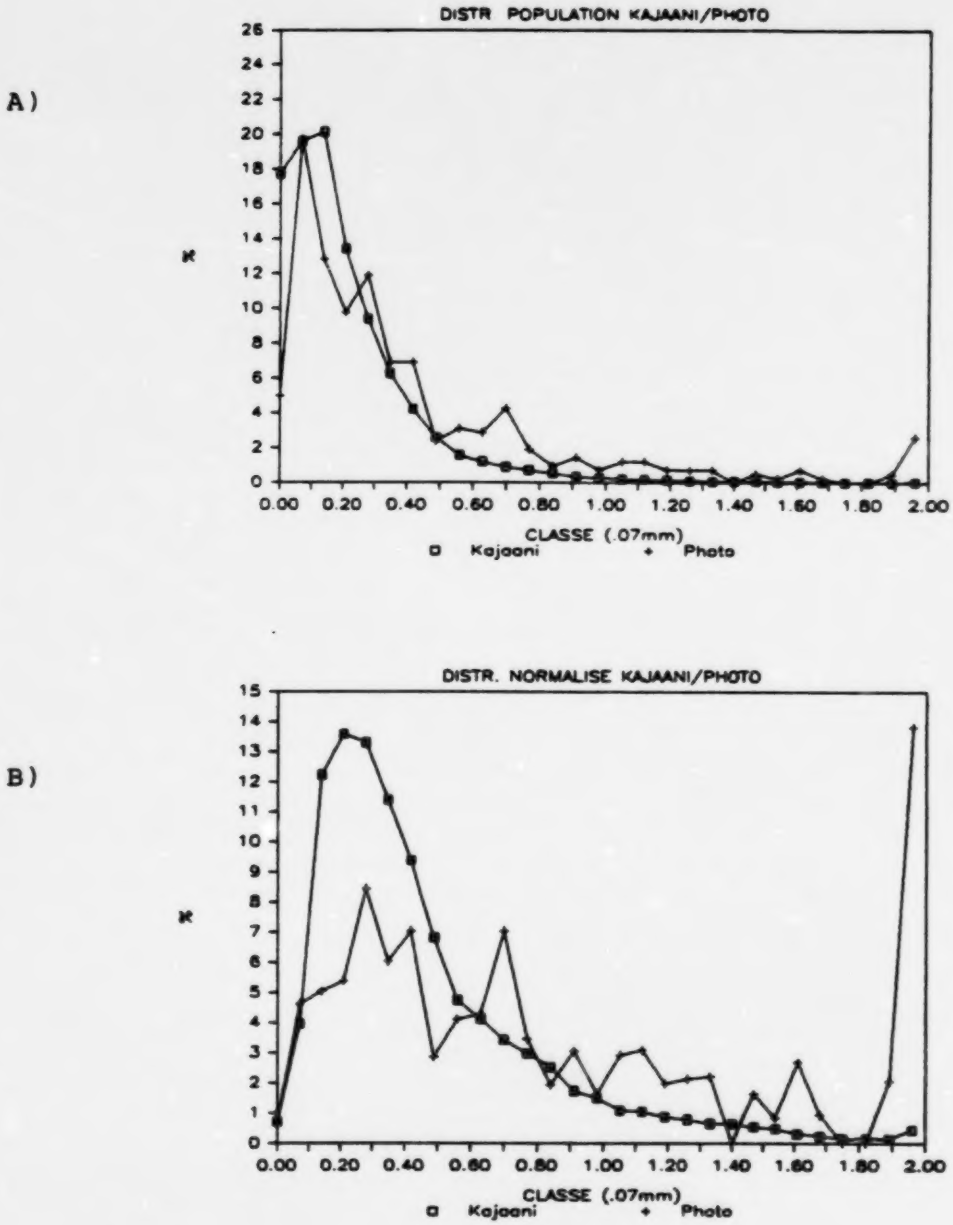


FIG. XI.2f) Comparison between KAJAANI (□) and optical microscopy (+) population length distributions of wet-milled Woodsreef 10-mesh open fibers, by .21 mm classes

CASSIAR DRY-MILLED AZ FIBERS (Open)

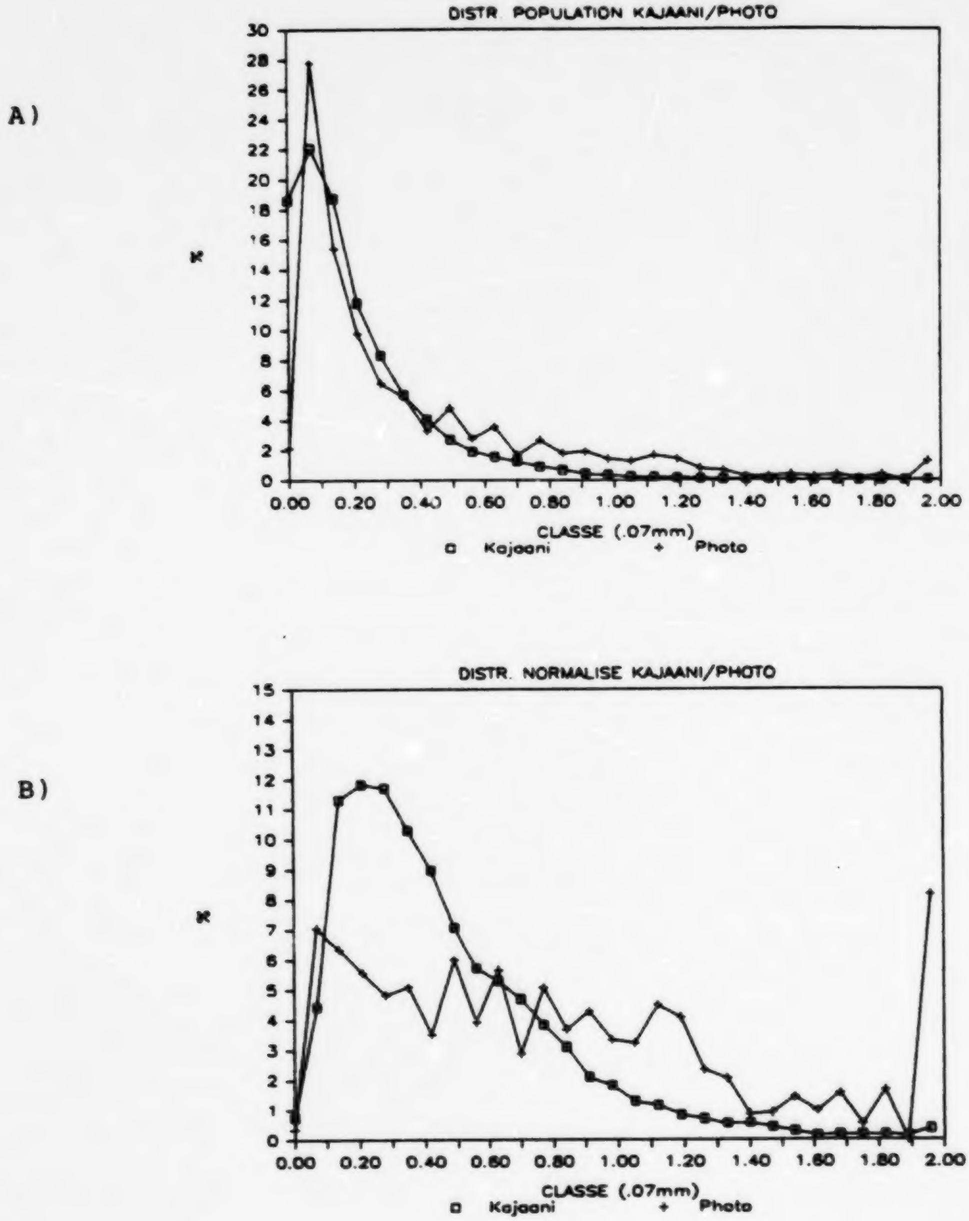


FIG. XI.2g) Comparison between KAJAANI (□) and optical microscopy (+) population length distributions of dry-milled AZ opened fibers, by .21 mm classes

CASSIAR DRY-MILLED AY FIBERS (Open)

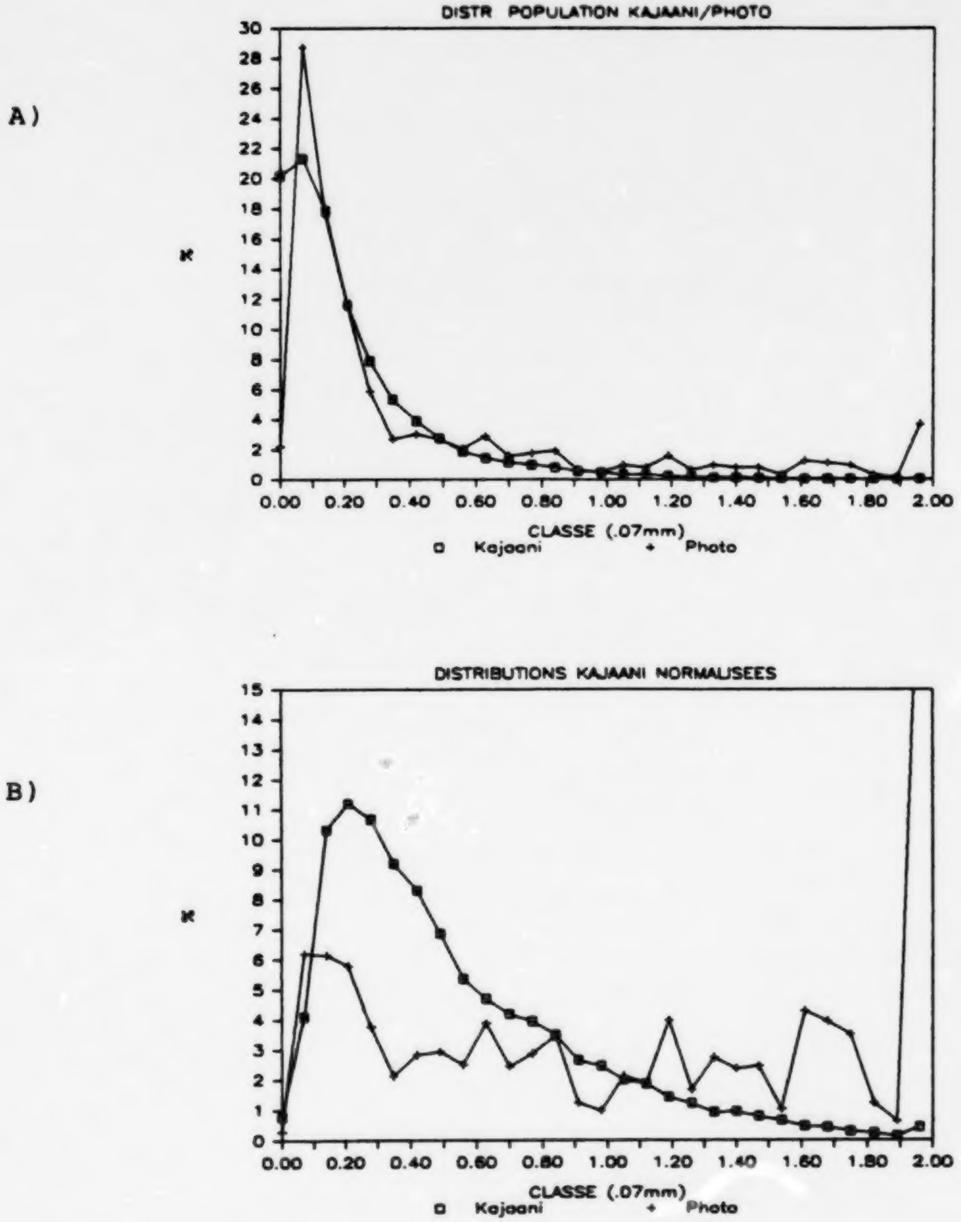


FIG. XI.2h) Comparison between KAJAANI (□) and optical microscopy (+) population length distributions of dry-milled AY opened fibers, by .21 mm classes

CASSIAR DRY-MILLED AX FIBERS (Open)

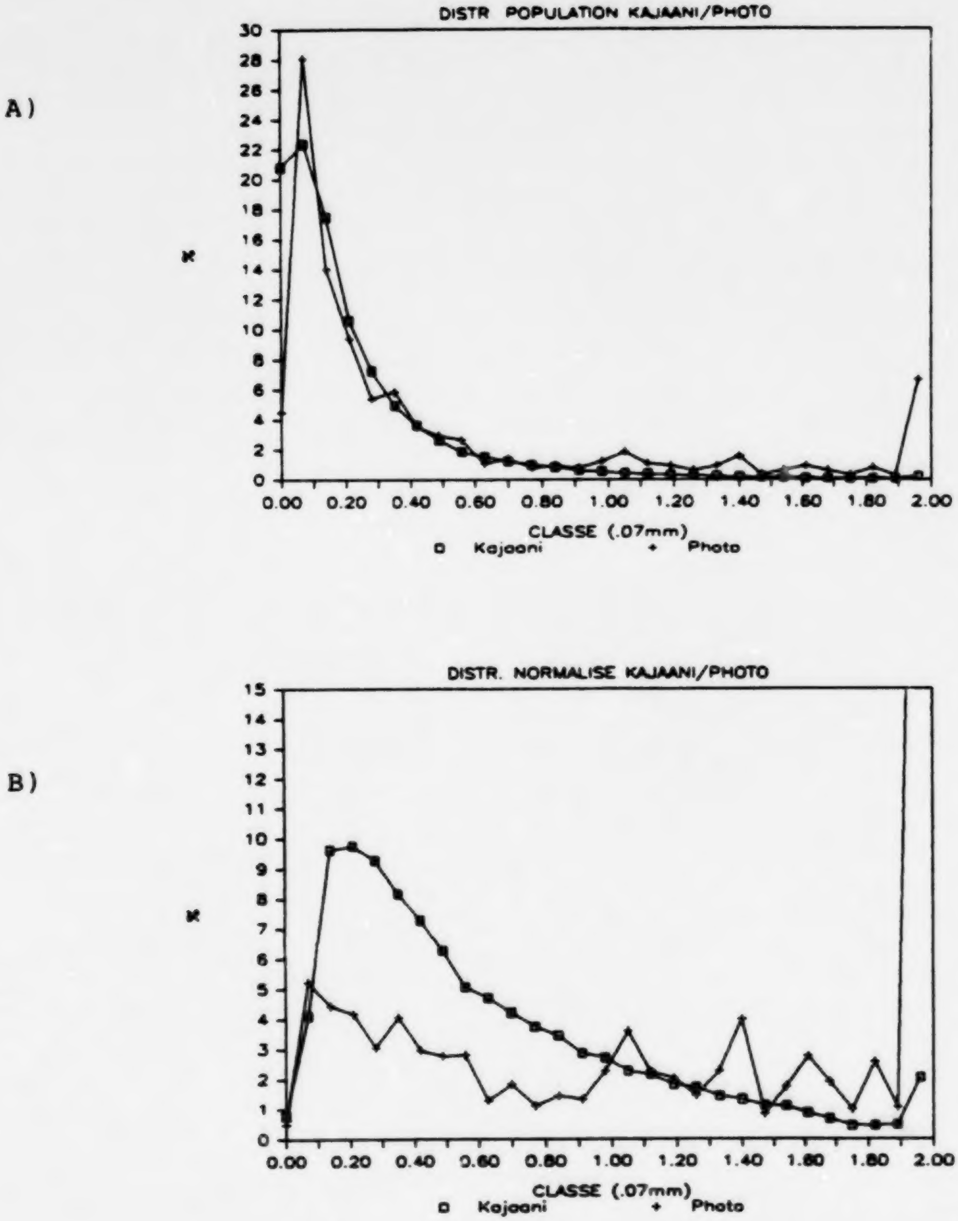


FIG. XI.2i) Comparison between KAJAANI (□) and optical microscopy (+) population length distributions of dry-milled AX opened fibers, by .21 mm classes

TABLE XI.1: Population and normalized mean lengths of wet- and dry-milled fibers (bulk and Pallmann opened), obtained from a Kajaani Length Analyser and through optical microscopy (OM). All values are in mm

Fiber types	Population		Normalized	
	Kajaani	OM	Kajaani	OM
Bulk (normal) fibers				
Wet-milled				
Cassiar S	.248	.347	.472	.537
" M	.468	.629	.750	.983
" L	.564	.774	.970	1.148
" XL	.572	.749	.940	1.176
" FEED	.407	.624	.657	1.037
Woodsreef 10-mesh	.233	.498	.440	1.172
Dry-milled				
Cassiar AZ	.244	.334	.548	.848
" AY	.268	.467	.622	1.168
" AX	.288	.409	.740	1.218
Pallmann opened fibers				
Wet-milled				
Cassiar S	.193	.380	.383	.646
" M	.360	.596	.583	.911
" L	.403	.666	.693	.993
" XL	.383	.727	.707	1.108
" FEED	.313	.522	.527	.838
Woodsreef 10-mesh	.210	.443	.423	.932
Dry-milled				
Cassiar AZ	.217	.421	.443	.900
" AY	.230	.486	.500	1.263
" AX	.243	.557	.580	1.408

The mean length values computed by our Kajaani FS-100 instrument is incorrect; values are between 25 and 50% shorter than they should be based on the distributions.

Visual inspection of the OM and Kajaani distributions, and comparisons among the mean length results (Table XI.1) lead to the following conclusions.

- Fiber length population distributions obtained from the Kajaani FS-100 analyser match the optical microscopy distributions rather well particularly in the case of wet-milled fibers; the Kajaani distributions are smoother (more regular), which is expected in view of the larger number of fibers counted in the Kajaani measurement, i.e., approx. 3 000 in Kajaani vs approx. 500 in OM.

- As noted earlier, the mean fiber length values obtained from Kajaani mesurements were found systematically lower (between 25-50% in most cases) than those obtained from OM data. This was shown to be a computation problem with our instrument.

- From the normalized length distributions, which emphasize differences between OM and Kajaani data, it is readily seen that:

  - the agreement is much better for wet-milled fibers and
  - the Kajaani apparently overestimates the shortest fiber classes.

The latter effect may be understood as due to either:

- non fibrous dust or grit particles which can trigger the optical detection and thus be counted in the shortest fiber classes;

- very thin long fibers (with diameters lower than the resolution limit of the Kajaani) are not "seen", while they are counted in optical microscopy measurements;

- curved fibers being measured only in part, i.e., only a fraction of the actual fiber length being "visible" to the Kajaani.

- Since the apparent Kajaani overestimation of short classes appears more severe with dry-milled fibers, we conclude that its main cause is contributions from dust and grit particles.

Before concluding this relative assessment, it is useful to recall that: 1) the OM measurements which may be considered "absolute length measurements" had a resolution limit of 2 μm in both fiber lengths and diameters and 2) grit and dust particles were not included in the OM measurements. In view of this and other remarks made above, the following summarizing statements seem appropriate:

- The Kajaani FS-100 yields a precise fiber length distribution with all fiber grades examined; the distribution will generally tend to be slightly skewed towards the shorter classes;

- Provided the dust fraction (very short fibers or particles) can be measured independently, the Kajaani analyser, or other instruments working on similar principles, should be extremely useful for quantitative length measurement and quality control on chrysotile fiber products.

XI.2 - Comparison of direct length measurement methods with Bauer-McNett (BMN) classification

This section deals with a central issue of the present study, attempting to answer the following questions:

- How accurately does the BMN classification separate fibers of specified lengths?

- Could a straightforward length measurement technique such as the Kajaani Fiber Length Analyser be substituted for the BMN test?

To provide quantitative answers to those questions, various correlations of the optical microscopy, Kajaani and Bauer-McNett data were pursued, taking into account that:

- OM data provide both the length and the diameter of the fibers;
- Kajaani data provide only the length of the fibers;
- BMN data give weight fractions collected on a set of consecutive screens: 4, 14, 35 and 200-mesh.

It is thus immediately apparent that OM data will enable calculation of fiber volume and weight (thus weight fractions) to compare with BMN results. On the other hand, Kajaani data can only be related to BMN data, if the fiber diameters are known by other means, or assumed. The following sections attempt to reconcile these three approaches to fiber length measurements.

XI.2 a - Direct comparison of OM and BMN results

We illustrate in Fig. XI.3 BMN data (bar chart) for the Cassiar wet- and dry-milled fiber products. The BMN classification was carried out only on bulk fiber products (BMN results for Pallmann opened fibers would also have been interesting).

Using the length and diameter data available from OM for each fiber, we computed a "reconstituted" BMN classification, assuming that the BMN screen apperture size determines the longest fiber passing through the screen. The fibers measured by OM are thus distributed into length classes with cut-off lengths at 75 μm (+200), 425 μm (+35), 1180 μm (+14) and 4750 μm (+4).

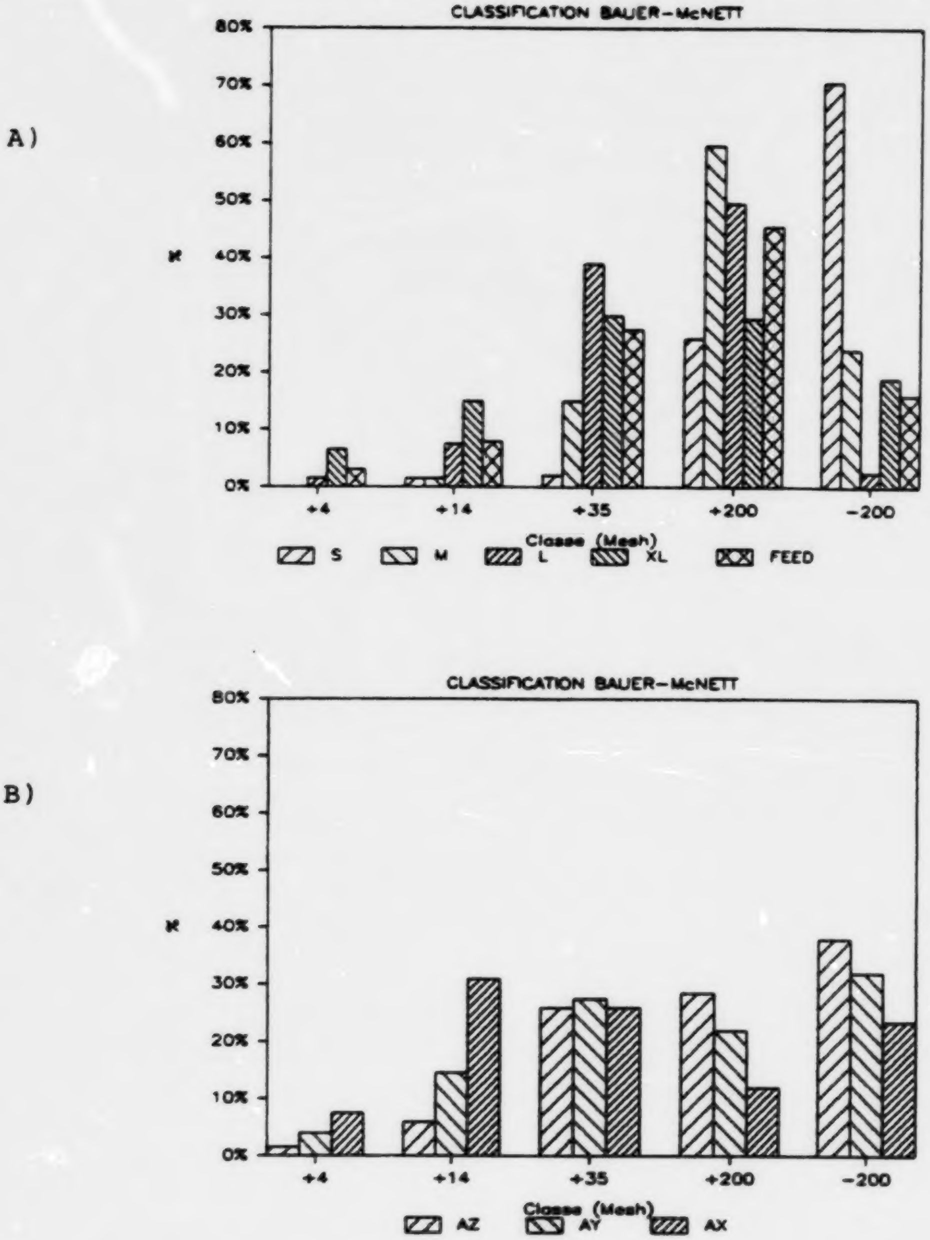


FIG. XI.3 Bar chart representation of the Bauer-McNett results for wet- and dry-milled Cassiar fibers

A) Wet-milled fibers
B) Dry-milled fibers

The BMN classifications simulated from optical microscopy measurements are illustrated in Fig. XI.4(a-b); a and b illustrate simulations carried out from OM data respectively from bulk and Pallmann opened fibers. Comparison of the true and simulated BMN charts show the following:

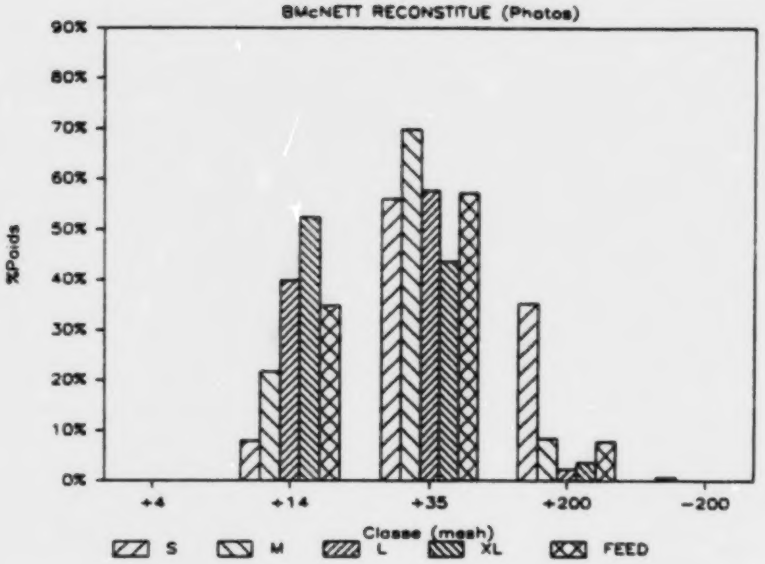
- a severe underestimation of the -200-mesh fraction in the simulated BMN; this may be expected since non fibrous dust particles were not included in OM measurements;
- an underestimation of the +14-mesh fraction in most cases, probably due to the small number of large fibers counted in OM measurements (this can also arise from other effects as described below);
- in a given BMN fraction, the relative ordering of different fibers (e.g., S, M, L and XL) is reasonably predicted in the simulated BMN (especially with wet-milled fibers);
- in the case of dry-milled fibers, the BMN charts simulated using the OM data on Pallmann opened fibers show improved agreement with the experimental BMN data;
- consequent to the underestimation of the -200 and +14-mesh fractions in the simulated BMN fractions, the intermediate classes are overestimated (the total must equal 100%).

Keeping in mind the limitations mentioned above on the OM measurements, the quantitative comparison of experimental and simulated BMN length classifications provide valuable insight into the significance of BMN data. To further assess the latter in relation to true fiber lengths, obvious physical or hydrodynamic effects must be acknowledged.

First, fibers longer than the size of the screen apperture can pass through the screen, thus increasing the shorter BMN fractions. This is clearly evidenced in the case of the wet-milled S product for which the large -200-mesh fraction (∿ 70%) mostly contains fibers longer than 100 μm (200-mesh corresponds to 75 μm).

CASSIAR FIBERS (Bulk)

A)



B)

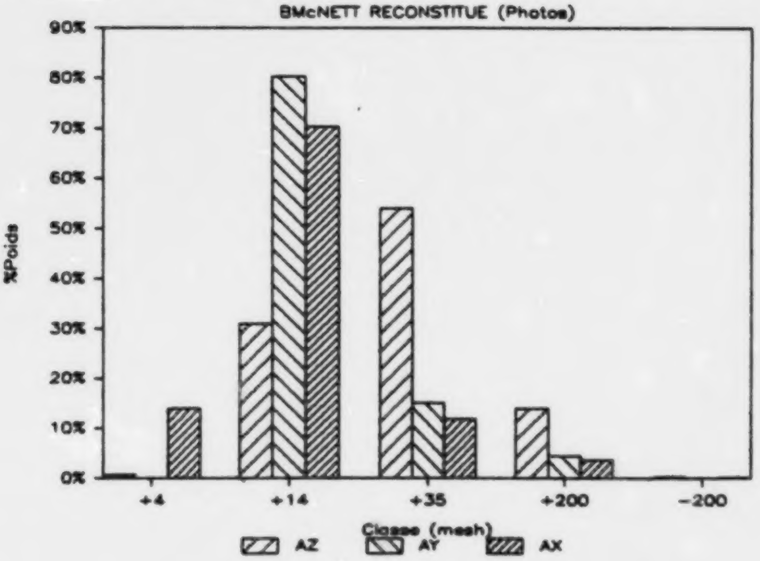
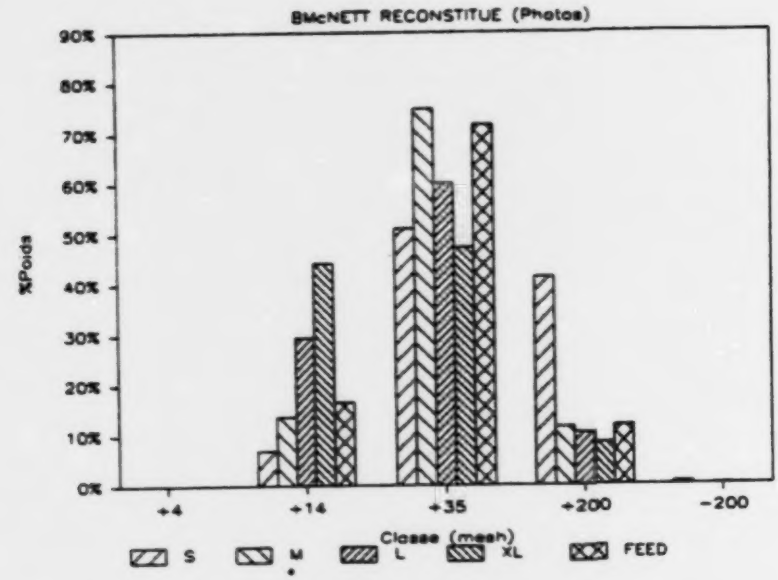


FIG. XI.4a) Bauer-McNett classification of Cassiar wet- and dry-milled fibers "reconstructed" from the fiber lengths and diameters measured by optical microscopy. The screen appertures were used as length cut-off points in the OM distribution

A) Wet-milled fibers
B) Dry-milled fibers

CASSIAR FIBERS (Open)

A)



B)

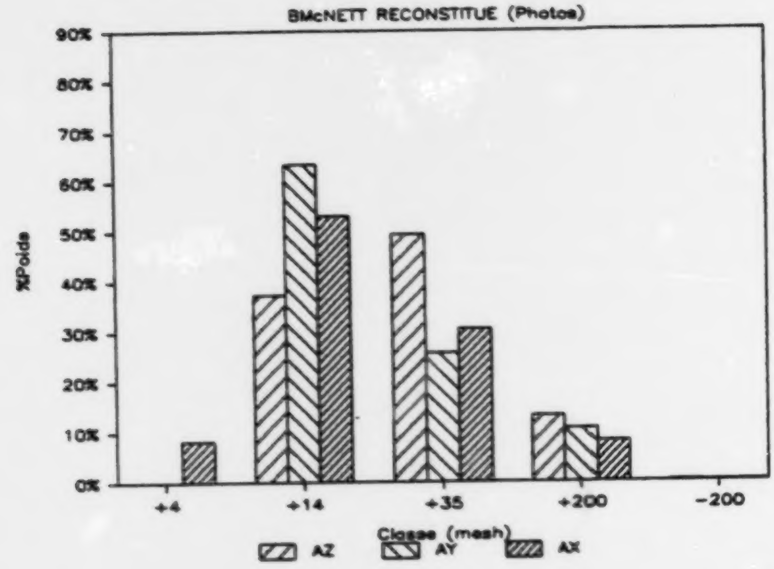


FIG. XI.4b) Bauer-McNett classification of Cassiar wet- and dry-milled Pallmann open fibers "reconstructed" from the fiber lengths and diameters measured by optical microscopy. The screen appertures were used as length cut-off points in the OM distribution

A) Wet-milled fibers
B) Dry-milled fibers

Second, in aqueous slurries, fibers tend to cling together and this effect will increase the longer BMN fractions. Based on our comparison of BMN and OM data, such effects would be particularly important with dry-milled fibers; this is not unreasonable since wet-milled fibers have already undergone attrition and conditioning in water prior to the BMN test.

Consequently, based on simple physical-hydrodynamic arguments, and on detailed comparisons with optical microscopy data, the Bauer-McNett test appears far from ideal for true fiber length classification.

To match BMN and optical microscopy data, we have inverted the procedure, that is: using the OM data, we determined the "effective screen apperture size", i.e., the true ranges of fiber lengths in each BMN class. This was carried out for each type of fiber examined here, leading to a range of "effective apperture sizes" for each fiber class. Taking the mean values of these "effective apperture sizes", it was possible to reconstruct BMN charts for the various fibers in better agreement with experimental results (as it should since the procedure is circular).

XI.2 b - Comparison of Kajaani and BMN classification data

As noted earlier, the Kajaani analyser only yields fiber length data; thus other measures of fiber diameters must be used to calculate weight fractions from the Kajaani length distributions. In the present case, fiber diameters are available from optical microscopy measurements as reported earlier.

To relate Kajaani and BMN data, we examined the diameter of fibers (optical microscopy) in fiber subgroups corresponding to BMN fractions. The mean fiber diameters found in each BMN fraction are plotted in Fig. XI.5a,b.

CASSIAR FIBERS (Bulk)

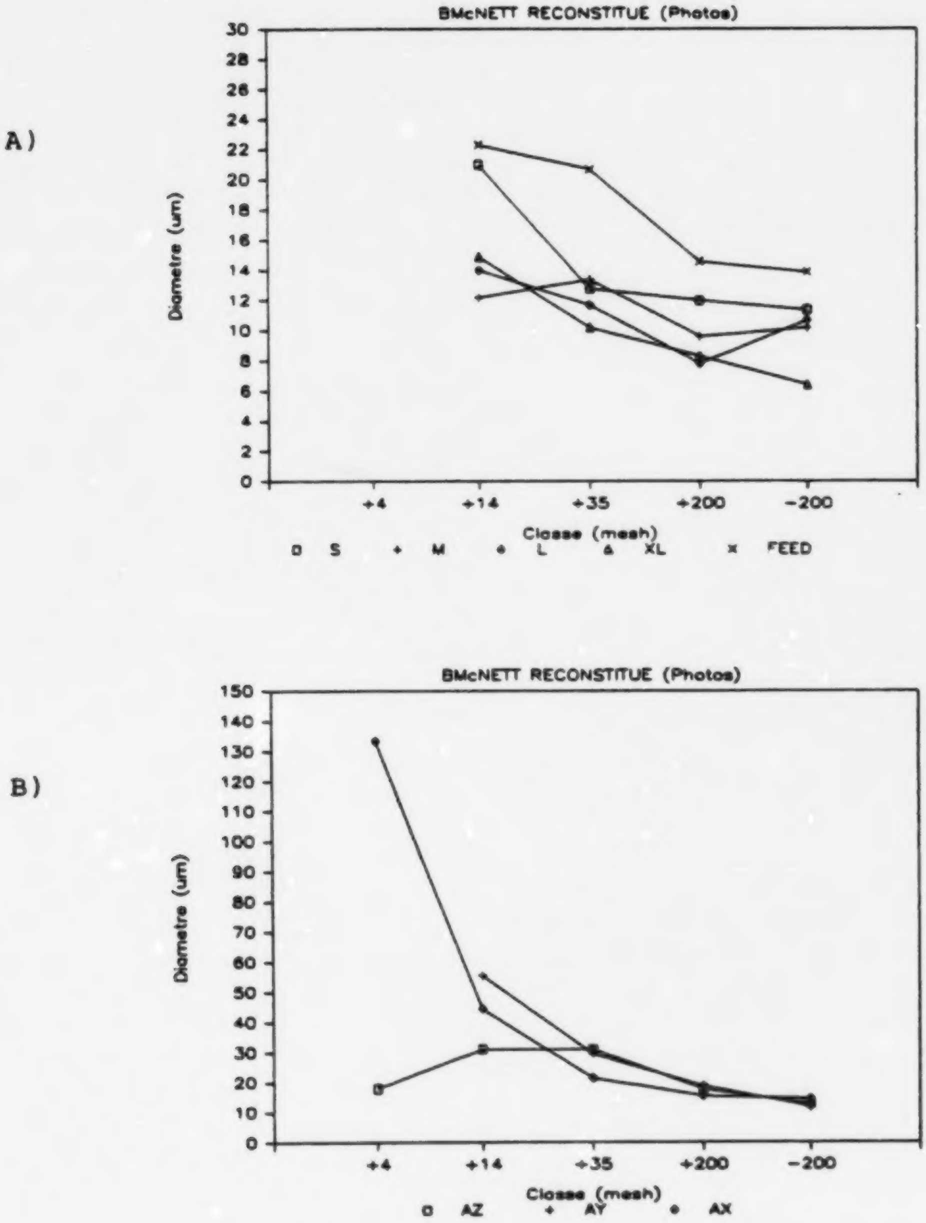


FIG. XI.5a) Average diameters of Cassiar wet- and dry-milled bulk (normal) fibers by length classes corresponding to Bauer-McNett fractions.

A) Wet-milled fibers
B) Dry-milled fibers

CASSIAR FIBERS (Opened)

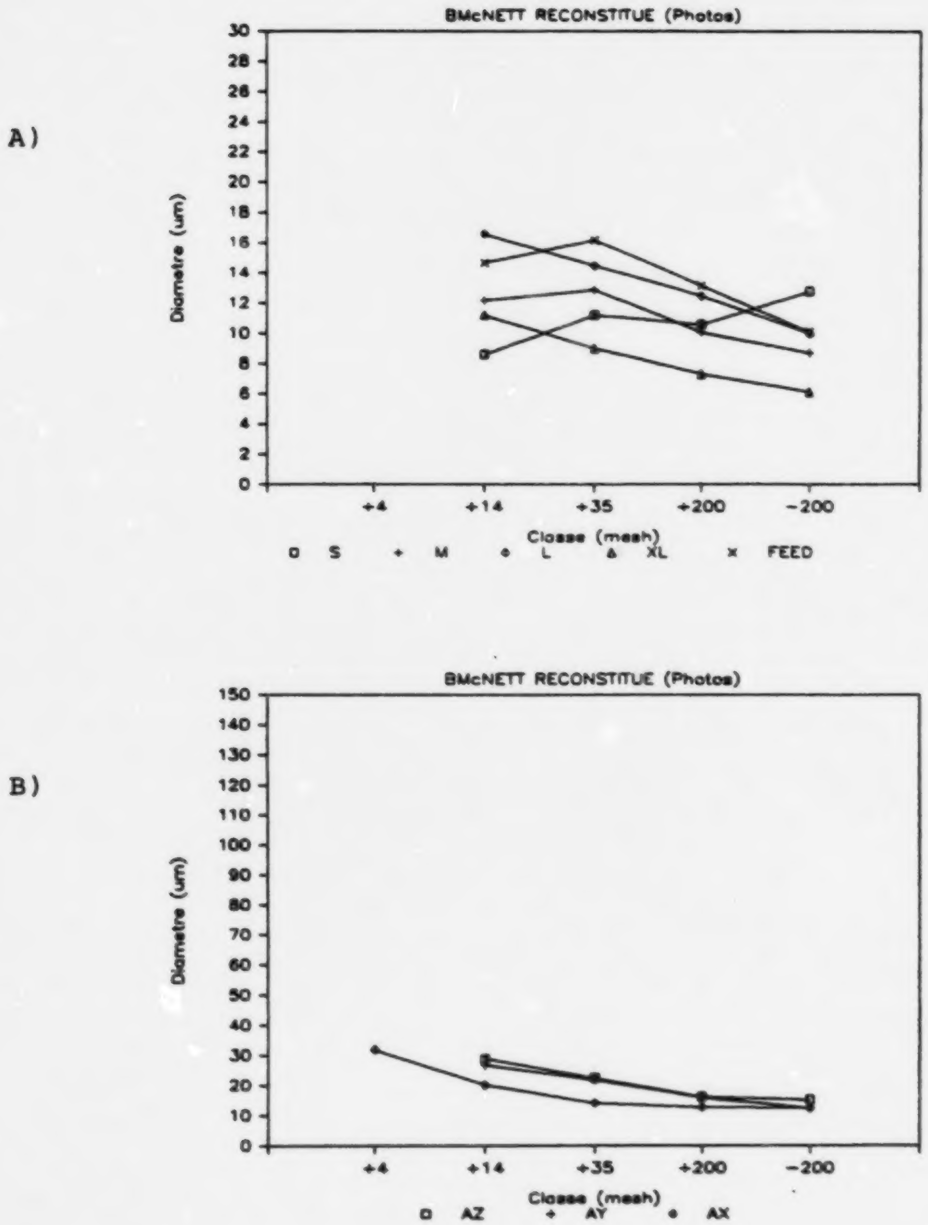


FIG. XI.5b) Average diameters of Cassiar wet- and dry-milled Pallmann opened fibers by length classes corresponding to Bauer-McNett fractions

A) Wet-milled fibers
B) Dry-milled fibers

Fig. XI.5a shows that with bulk fibers, the average diameters generally decrease in going from the long (e.g., +14) to the short (e.g., -200) BMN fractions (the average diameters in the +4 fractions are not reliable because of the small number of fiber measured by OM in these groups). In order to reconstruct a BMN fraction chart from Kajaani data, it would thus be required that we assign the fibers in each fraction the different diameters shown in XI.5a. The usefulness of such a procedure would be limited in practice, since average diameters could vary substantially in production processes.

On the other hand, it was noted earlier (Section VII.2.) that following Pallmann opening, the average fiber diameters within a narrow size range; Fig. XI.5b further shows that the diameters of opened fibers in the various BMN fractions exhibit only weak variations (again, average diameters decrease in going from the +4 to the -200 BMN fraction). The latter finding is of considerable importance; it suggests that upon prior Pallmann (or possibly other techniques) opening, which could be done in a standardized way, a single average diameter could be assigned to all classes.

Assuming a constant fiber diameter for the BMN subgroups of a given fiber type and the Kajaani normalized fiber length distributions of wet- and dry-milled Pallmann opened fibers, we calculated the BMN weight fractions shown in Fig. XI.6. The actual value chosen for the diameter is irrelevant; the critical assumption is the it should be the same in all BMN fractions.

Comparison of the predicted and experimental BMN results lead to the following significant observations:

- The predicted -200-mesh fraction is lower than the measured one; this is readily understood since: 1) the Kajaani cannot "see" micron-size particles or fibers and 2) the BMN -200 class contains fibers much longer than the 200-mesh apperture size.

CASSIAR FIBERS (Open)

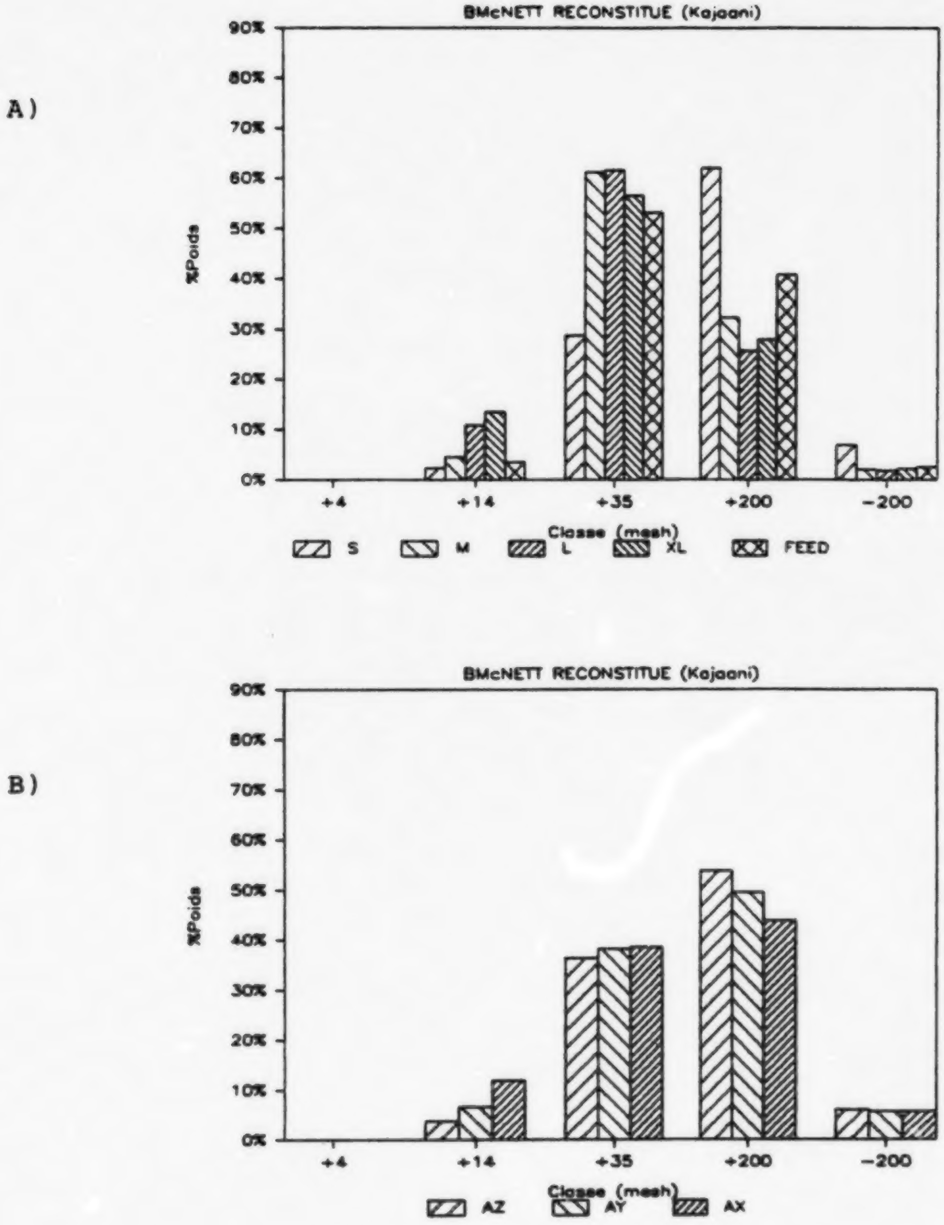


FIG. XI.6 BMN weight fractions reconstructed from KAJAANI normalized length distributions of Pallmann opened fibers, assuming constant fiber diameters in all BMN groups

A) Wet-milled fibers
B) Dry-milled fibers

- The reconstructed BMN charts show no +4 fraction; this is simply due to the fact that the coarse fiber bundles or fiber aggregates (from fiber-fiber adhesion) will not go through the Kajaani capillary.

- In the other BMN fractions (+200, +35, +14) the predicted weight fractions are higher than the observed ones (as required for 100% sum); the relative ordering in each BMN fractions, for example the relative weight fractions of AX, AY and AZ in each of the +14, +35 and +200 groups are reasonably well predicted.

The procedure followed here to relate BMN and Kajaani data (on Pallmann opened fibers) rests on a single assumption, namely: the constancy of fiber diameters in the various classes. We submit that this procedure yields reliable fiber length measurements at least in the BMN fractions which are most relevant to the A/C industry: +14, +35 and +200-mesh fractions.

As specific answers to questions raised at the beginning of this section, the data and correlations discussed above provide the following:

- The results of Bauer-McNett classification on bulk commercial fibers should not be interpreted as fiber length distributions in classes corresponding to screen apperture size; the length classification in a BMN test appears to overestimate both the fractions of shortest (-200-mesh) and longest fibers (+4).

- A reliable fiber length distribution can be obtained with a Kajaani FS-100 Fiber Length Analyser for the industrially important lengths which correspond to the BMN fractions +14, +35 and +200.

- A valid quality control, or process monitoring, application of the Kajaani Analyser will require a preliminary fiber opening treatment, first to ensure removal of the larger bundles which would clog the capillary cell and, second, to reduce the average fiber diameter to a constant value. The full characterization of a bulk fiber product will require an independent measurement of fine dust particles and grit which cannot be reliably assessed with the Kajaani analyser.

XII - SELECTED SURFACE CHARACTERIZATION RESULTS

In this section we describe methodologies and results which were pursued to provide information on the microstructure and surface properties of the various fiber products examined.

The first two types of results presented are BET nitrogen (N_2) surface area and mercury porosity; both types of data are obtained from dried fibers and both relate to the surface area and porosity of the fibers (hence, in principle, to their degree of opening).

The third category of results describe the adsorption of dye molecules (large organic molecules) onto selected fiber products. Dye adsorption is measured directly in aqueous fiber slurries and it also relates to surface area and surface micro-porosity of the fibers. The latter approach was explored mainly with the goal of developing methods for direct measurement of fiber surface area and extent of fiber opening in aqueous slurries.

XII.1 - Nitrogen surface area results

The BET nitrogen surface area of the various Cassiar wet- and dry-milled fiber products are collected in Table XII.1. The dry-milled fibers AX, AY and AZ exhibit surface areas comparable to those of other dry-milled products of similar grades. The wet-milled fibers (M, L, XL and FEED) have, on the average, a slightly higher surface area than the dry-milled fibers; the S fibers exhibit, on the other hand, a distinctly higher surface area than all other products ($\sim$50% greater).

TABLE XII.1: Nitrogen gas surface area of Cassiar wet- and dry-milled fibers

Sample		Surface area (m^2/g)
Wet-milled	S	29.5
	M	20.4
	L	18.0
	XL	21.1
	FEED	18.3
	FEED (Pallmann opened)	18.6
Dry-milled	AZ	18.3
	AY	15.8
	AX	17.8

The above results follow the same overall trend as those obtained from the standard RSA (Rapid Surface Area) data reported earlier in Table IX.3; the RSA values showed, however, much greater distinction between the wet- and the dry-milled fibers: RSA values of the M, L, XL and FEED fibers were approximately twice those of the AZ, AY and AX fibers.

At this point it is useful to recall that the BET surface area measurement will include contributions from the fine dust fraction as well as from the fibers; in fact the dust fraction with high surface area can be a very substantial part of the total surface area measured. This, we believe, explains why the dry-milled fibers have N_2 surface areas comparable to the wet-milled (M, L, XL and FEED) fibers, while the RSA values differ considerably: the dry-milled products contain much more dust which raises their N_2 surface areas.

The intrinsic difference in the sensitivity of the RSA and N_2 surface area methods towards the dust fraction should be explored in more details as a potential means to evaluate dust fractions of fiber products.

XII.2 - Mercury porosity

The penetration of liquid mercury in a porous solid, measured as function of applied pressure, yields information on pore volumes and effective diameters. Preliminary experiments were carried out to assess the usefulness of this technique in characterizing the degree of opening of chrysotile fibers.

Typical mercury porosity results are shown in Fig. XII.1 for selected dry- and wet-milled fibers; the results are shown as cumulative pore volume plotted against decreasing effective pore radii (i.e., as the pressure is increased, mercury will penetrate the smaller pores). Two important observations may be noted from these data:

- first, most of the pore volume corresponds to pores having radii in the range 0.1-3.0 µm;

- second, Pallmann opened fibers exhibit about twice the porosity of the other dry-milled (AY, 4T) or wet-milled fibers (M); we may also note that the wet-milled fibers have pore volumes approximately 10% larger than the dry-milled fibers.

The significance of these results can be understood if we relate the measured pore radii to the diameter of the fibers and the expected interstices in a compacted sample of chrysotile fibers.

The average diameters of chrysotile fibers (wet- or dry-milled) fall in the range 10-25 µm. We would thus expect that random packing of such fibers will leave voids with effective radii in the order of 1 or a few µm. Hence, the finding of most of the pore volume near 1 µm pore radii is consistent with the physical nature of fiber samples.

MERCURY POROSITY OF SELECTED FIBERS

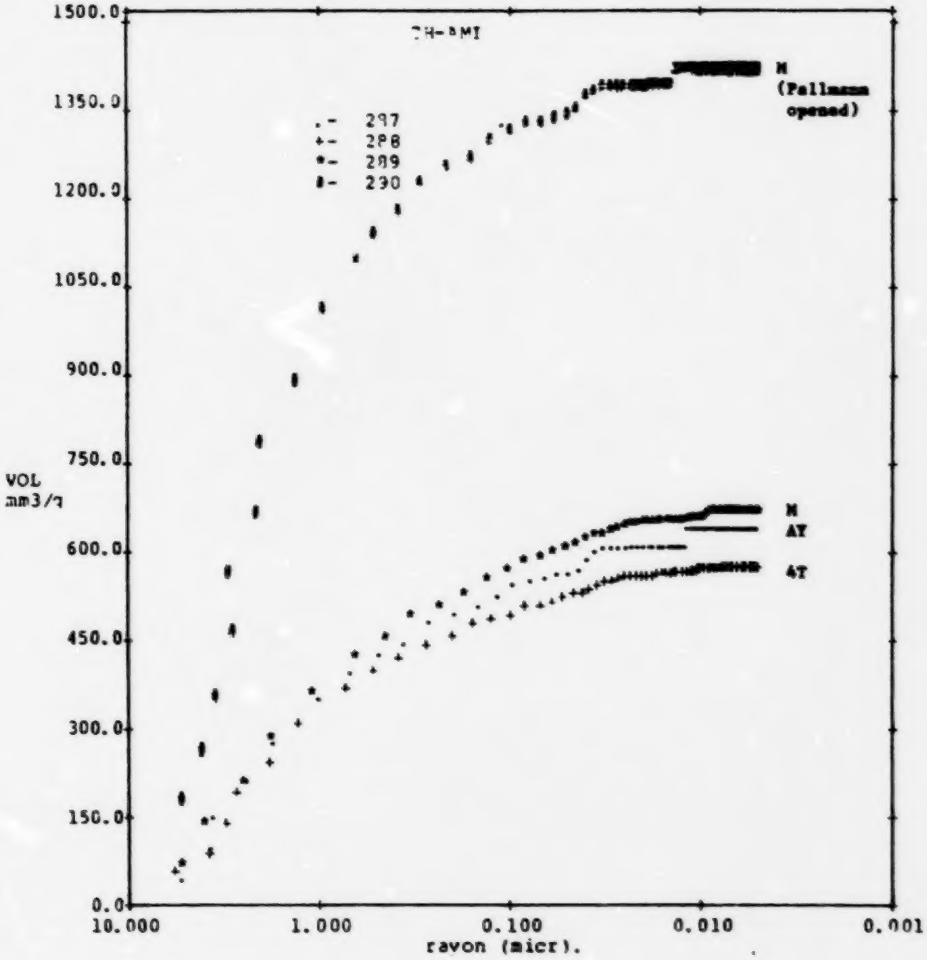


FIG. XII.1 Integrated pore volume (mm^3/g) as function of pore effective radii for selected Cassiar and Quebec 4T fibers

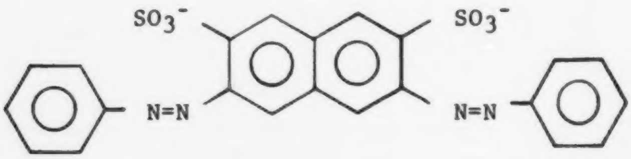
The large pore volume increase found after Pallmann opening is also consistent with our previous observations that Pallmann opening introduces "kinks", "bruises" and "partial splitting" of the fibers. These effects will further increase the inter-fiber voids, as one would anticipate from simple fiber packing considerations (i.e., bent nails will not pack as tightly as straight ones). While our observations rest on very limited data, the magnitude of the changes observed upon Pallmann opening suggests that this type of measurement will be ideally suited to measure the extent of fiber opening.

Finally, the slightly higher mercury porosity found with the wet-milled fibers (M) compared to dry-milled fibers (AY or 4T) is also consistent with the view that wet-milled fibers are more opened than the dry-milled products (the "loosening" of fibers in the fiber bundles woudl lead to crevices in the size range 0.1-1.0 μm).

XII.3 - Wet surface area from dye adsorption in aqueous slurries

It is well known that chrysotile strongly adsorbs numerous organic dyes from aqueous solutions; we have explored the use of this phenomena to gain direct access to the surface area of chrysotile fibers in aqueous slurries.

Various dye compounds were selected for preliminary studies to identify one particular compound having the following desirable behavior: high affinity for chrysotile, chemically stable when adsorbed and only adsorbing as monolayers. The anionic dye "Acid Black" (also called "Naphtol Blue" or "Amido Black T") was selected as best candidate according to the above criteria.



(Acid Black)

In basic solution the dye molecule is negatively charged and this favors its adsorption on the positively charged chrysotile surface.

A measurement of the amount of dye absorbed as function of increasing dye concentration in solution yields adsorption isotherms as shown in Fig. XII.2 for selected fiber types.

These data were analysed as Langmuir (or BET type 1) isotherms which are described by the following equation:

$$\frac{C}{n_{ads}} = \frac{C}{n_s} + \frac{1}{n_s b}$$

where C is the solution concentration of the dye and n_{ads} is the amount (moles) of dye adsorbed on the fibers at that concentration; n_s is the amount adsorbed at full monolayer coverage of the surface (surface saturation) and "b" is a constant. Fitting the experimental data to this equation yields two important parameters:

n_s : a dye quantity related to the fiber surface area;
b : a constant related to the affinity of the dye molecules for the fiber surface.

The n_s and b values calculated from the data shown in Fig. XII.2 are collected in Table XII.2

ADSORPTION ISOTHERMS

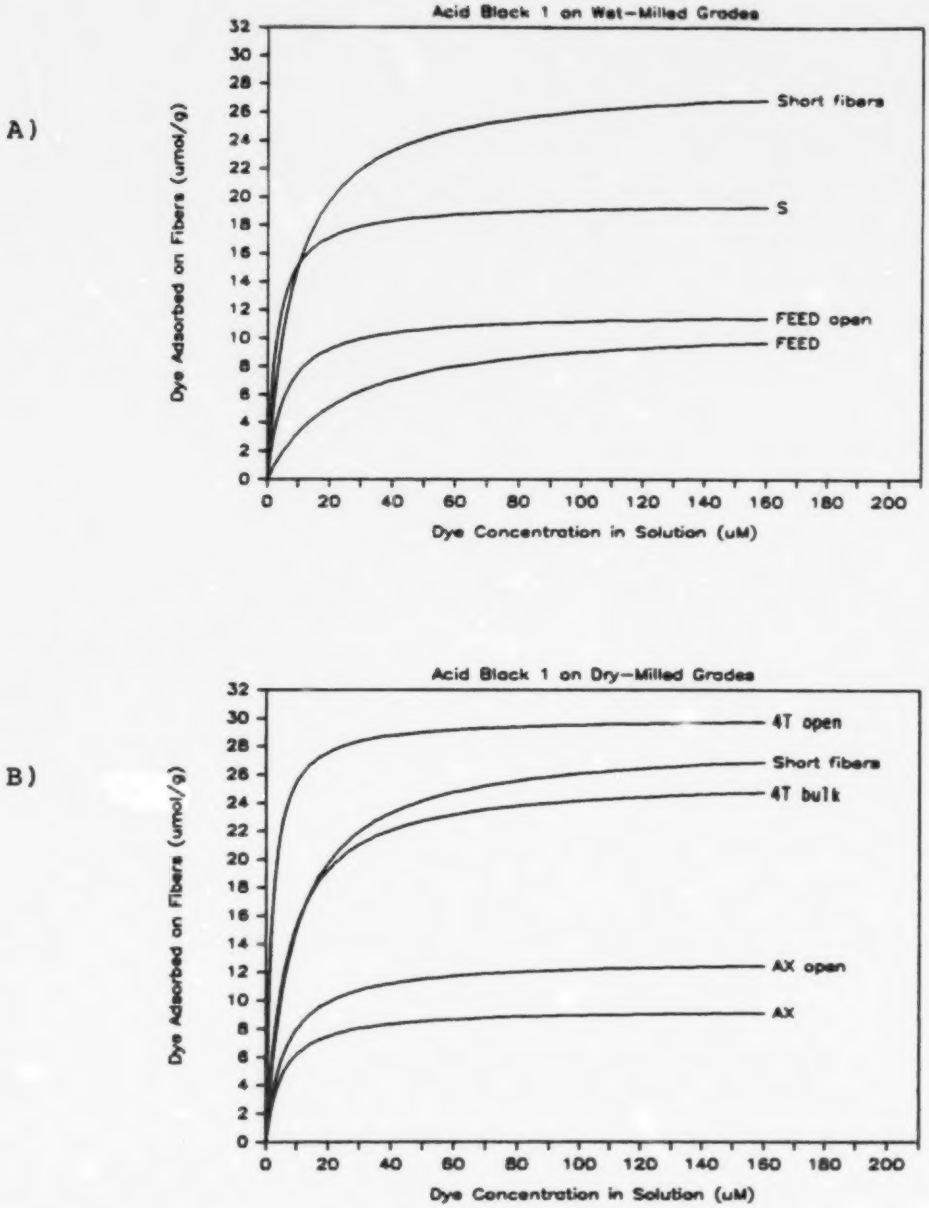


FIG. XII.2 Adsorption isotherms for Acid Black 1 on chrysotile fibers in aqueous slurries

TABLE XII.2: Adsorption parameters for Acid Black on selected chrysotile fibers

Fiber type	n_s (μmol/g)	b (1/(μmol*1))
Cassiar S	19.65	0.346
Cassiar FEED	11.14	----[2]
Cassiar FEED opened	11.83	0.180
Cassiar AX	9.42	0.194
Cassiar AX opened	12.94	0.163
Quebec 4T	25.77	0.149
Quebec 4T opened	30.03	0.563
Quebec "short fibers"[1]	28.33	0.115

1 The "short fibers" are a colloïdal fraction obtained by wet extraction of a Quebec 4T product.
2 Not determined with sufficient accuracy.

Within the limited set of data available at present, the following trends appear significant:

- from the n_s values, the Cassiar wet- and dry-milled fibers (FEED vs AX) exhibit comparable surface areas, which is consistent with our similar finding from nitrogen adsorption;
- the S fibers exhibit an n_s value approximately twice the value found for the FEED fibers,which is again consistent with nitrogen adsorption data;
- the Quebec 4T fibers show the highest n_s values which is perhaps a consequence of higher dust content (not measured);

- the affinity parameters, (b), vary considerably and this may reflect, in part, experimental difficulties; however, visual inspection did show that the adsorbed color intensity varied among the different types of fibers, and also comparing fibers and dust particles; non fibrous dust particles were found to adsorb the dye molecules with higher affinity than the chrysotile fibers.

The results reported here thus support the following conclusions:

- fiber surface areas can be measured directly in aqueous slurries and yield data comparable to BET nitrogen surface areas;

- in view of the varying affinities of the dye molecules for different types of chrysotile fibers, the dye adsorption method will require calibration for each specific fiber type.

XIII - CONCLUSIONS

The data collected and analysed in the course of this study yielded considerable new information and insight on dimensional and morphological characterization of chrysotile asbestos fibers. This information provides an adequate basis for resolving the key issues raised by the contractor at the onset of this project. We conclude this report by a brief review of our main findings concerning, first, specific problems related to the Cassiar wet- and dry-milled fiber products and, second, the more general questions of fiber characterization for fiber quality control or monitoring of fiber milling processes. Detailed conclusions and recommendations are given in the Executive Summary.

XIII.1 - Cassiar wet- and dry-milled fibers

The bulk wet-milled fibers (identified here as "FEED") prepared by the Cassiar process exhibit a number of distinctive technical properties; when compared under identical conditions to Cassiar dry-milled fibers, the wet-milled fibers show:

- lower dust and grit contents;
- lower magnetic rating (low magnetite content);
- higher fiber strength (FSU).

In addition, the wet-milled product (after drying) was found easier to open in a Pallmann mill, i.e., suggesting that the wet-milling process leads to local or micro opening of the fibril in the individual fiber bundles.

All the classified Cassiar wet-milled products (identified S, M, L, XL) with the exception of the shortest fraction (S) exhibit very similar properties (lengths, diameters, texture, magnetic rating, FSU); the S fraction although consisting almost exclusively of fibers shows distinctly lower FSU and higher magnetic rating. Interestingly, the wet classification process was shown to lead to significant opening of the bulk wet-milled product (FEED).

Comparison of the wet-milled Cassiar and Woodsreef products show that the average fiber length of the Cassiar bulk FEED fibers is approximately two fold greater than the Woodsreef 10-mesh fibers; the latter fall intermediate between the S and M fibers of Cassiar.

XIII.2 - Fiber characterization methods

The results of our study show that most standard asbestos testing methods, wet or dry, can be applied to wet-milled products; dry tests may be performed after drying (oven, microwave or freeze drying) of the wet-milled products and subsequent mild opening (e.g., Pallmann or willow). However, in the interpretation of the test results, due account must be given to the intrinsic differences between the wet- and dry-milled products: for example the wet-milled Cassiar S product contains mostly very short fibers and little dust: hence, the -200-mesh BMN fraction of this product must be viewed accordingly.

- A fiber length analyser designed for the pulp and paper industry, Kajaani FS-100 (and presumably other similar instruments), was found to rapidly yield (∿ 10 minutes) highly reproducible fiber length distributions.

- The Kajaani FS-100 fiber length distributions match those derived from optical microscopy measurements quite well, especially with wet-milled fibers; the Kajaani instrument tends to overestimate the short fiber classes, but this should not be a major drawback.

- Detailed analysis of optical microscopy, Kajaani and Bauer-McNett data indicates that the reliable range of chrysotile fiber length measurements with the Kajaani FS-100 is approximately 100 μm - 3000 μm (0.1 mm - 3 mm), i.e., corresponding approximately to the +200, +35 and +14-mesh groups of the Bauer-McNett classification.

The routine use of a Kajaani FS-100 (or other model) Fiber Length Analyser in fiber quality control, or monitoring purposes will require:

- a preliminary treatment to open the larger fiber bundles, first, to avoid instrument clogging and, second, to reduce the average fiber diameter to a near constant value;
- an independent method to evaluate the dust and grit contents.

If those conditions are met (constant average fiber diameter and independent measurement of dust fractions), our results show that the Kajaani analyser can yield adequate fiber length distributions which should be as pertinent to the user industry as is the BMN classification test with either wet- or dry-milled fibers. The constancy of average diameter is reasonably achieved by a simple fiber opening treatment (e.g., Pallmann, Dounce tube).

At present, we have no means of evaluating the dust content of fibers in a way comparable (relative ease and rapidity) to the fiber length analyser; a standard elutriation test could of course be performed in parallel to Kajaani measurements. Implementation of a fiber length analyser in fiber quality control may be envisaged, initially, as a parallel test to the Bauer-McNett classification, since statistical validation will require extended parallel testing. Simultaneously, other means for rapid quantitative dust contents of fiber products should be investigated.

Our attempts to develop a surface area measurement for fibers in aqueous slurries yielded highly promising data. Measurements of the absorption of organic dye molecules by wet fibers showed that both the surface area and the nature of the particules are important (i.e., surface area and surface affinity). The resolution of these two contributions to dye adsorption is possible, but would require considerable more data than the present study could provide, this should provide an alternative rapid method to evaluate both the surface area and the dust content. A full investigation with several carefully selected dyes should yield methods for rapid and simultaneous measurements of fiber surface area and dust contents.